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INDUSTRIAL WASTE SURVEY REPORT, HELD AT HERCULES POWDER COMPANY, NAVAL STORES DIVISION, HATTIESBURG, MISSISSIPPI. 13 FEBRUARY TO 20 APRIL 1951. STUDIES ON THE WASTES OF A WOOD NAVAL STORES PLANT

Virgil A. Minch, et al

Environmental Health Center
Cincinnati, Ohio

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Industrial Waste Survey Report

HERCULES POWDER COMPANY

Naval Stores Division

Hattiesburg, Mississippi

February 13 to April 20, 1951

Studies on the Wastes of a Wood Naval Stores Plant

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ABSTRACT

This survey was undertaken as a part of the Public Health Service program of industrial waste studies.

Studies were made of the main plant effluent and the various components that make up this waste and of the stump washer effluent, both of which discharge into the Bowie River. Special investigations were conducted on some of the more important plant processes and wastes.

Hercules Powder Company, Hattiesburg Naval Stores Division, operates 15 extractors to produce various grades of wood rosin, turpentine, pine oil, dipentene, and other terpene chemicals. An average of 800 tons of pine stumps are processed daily, from which 320,000 pounds of rosin, 4,000 gallons of turpentine, 2,400 gallons of pine oil, and 2,400 gallons of other liquid pine products, including dipentene, are produced.

All rosin thus produced is used in secondary plant units to make more valuable products. Secondary or specialized resin production averages 300,000 pounds daily. Wastes from these units contained 64 per cent of the total plant 5-day B.O.D.

The main plant effluent discharges daily 10,000 pounds of B.O.D., 5,500 pounds of suspended solids, and 12,000 pounds of dissolved solids. The 731 (disproportionation) plant is responsible for 46 per cent of the B.O.D. and 20 per cent of the total volatile solids found. The synthetics plant contributed 62 per cent of the ether-extractable material, 14 per cent of the B.O.D., and 12 per cent of the total solids found in the main plant effluent.

The stump washer effluent discharges 700 pounds of 5-day B.O.D., 33,000 pounds of suspended solids, and 7,000 pounds of dissolved solids daily.

Studies of the solvent and furfuraldehyde recovery units indicate that 62 % of the B.O.D. and 62-66% of the oxygen consumed is due to the presence of terpin hydrate in these wastes. Dissolved oils account for 67% of the B.O.D. and 71% of the oxygen consumed in the still house oil trap effluent.

Special phenol studies show that the plant discharges 14 lbs. daily. Of this total, the isomerization wash water contributes 7 lbs. and the synthetic plant 1 lb. A deficit of 6 lbs. daily was not found.

Grab samples from the main ditch show variations in B.O.D. concentrations of 190 to 1300 p.p.m., indicative of varied plant operations. Population equivalent of the total plant effluent was 62,900, based on 5-day B.O.D.

GENERAL INFORMATION

1. Organization of Survey

Arrangements for this co-operative project were made by officials of the Mississippi State Board of Health. The Environmental Health Center of the United States Public Health Service was invited to study naval stores wastes by the Southeast Drainage Basin office. Conferences were held by personnel of the Mississippi State Board

of Health, Environmental Health Center, and the Hercules Powder Company, to discuss results of previous studies, analytical procedures, and the conduct of the survey.

2. Dates

February 13 to April 20, 1951.

3. Personnel

The following personnel participated in the organization and conduct of the survey:

MISSISSIPPI STATE BOARD OF HEALTH

H. A. Kroeze, Director, Sanitary Engineering
C. M. Leadbetter, Sanitary Engineer

HERCULES POWDER COMPANY

L. C. Hall, Plant Manager
Frank Gardner, Research Chemist
Bruce W. Dickerson, Sanitary Engineer

UNITED STATES PUBLIC HEALTH SERVICE

Hayse H. Black, Senior Sanitary Engineer

UNITED STATES PUBLIC HEALTH SERVICE, MOBILE LABORATORY

Virgil A. Minch, Jr., Asst. Sanitary Engineer (R)
Albert L. Platz, Jr., Asst. Sanitary Engineer
Donald D. Gold, Sr. Asst. Sanitary Engineer
William W. Walker, Chemist
Carl N. Shadix, Chemist
Jesse Cohen, Chemist
Stephen D. Pinkoski, Chemist
Melvin D. Currey, Chauffeur-Mechanic

4. Purpose

This survey was conducted to determine the source, quantity, concentration and characteristics of process wastes resulting from the operation of a wood naval stores plant. This information will serve as an aid in determining the principal factors influencing the characteristics of a wood naval stores plant wastes. The results of this survey and similar studies will serve as a basis for preparation of an industrial waste guide on wood naval stores plants.

5. Location of Plant

The main offices of Hercules Powder Company, Naval Stores Department, are located at Wilmington, Delaware. The industrial plant, laboratories, and offices of the Hattiesburg Naval Stores Division, occupy approximately 240 acres of land along West Seventh Street in Hattiesburg, Mississippi.

6. History of the Naval Stores Industry in the United States

The production of naval stores can be traced back several hundred years. . Seagoing Phoenicians used rosin in the caulking of ships and ships' holds and for the treatment of rope. Records show that in 1608 tar, pitch, turpentine and rosin were made in Virginia and shipped to England. This cargo marked the first recorded shipment of industrial products from America to the Old World.

New England was the biggest producer in the New World until the late 1680's when settlers in North Carolina discovered that the

southern yellow or longleaf pine was a more abundant source of gum than the pitch pine of New England. By 1700 the production of naval stores was an important part of the economy of North Carolina.

The area of production of gum naval stores has shifted through the years. The Carolinas, once the largest producers, now account for less than one per cent of the total United States production. The change was brought about by the cutting of the virgin forests in those states without leaving seed trees for reproduction. The original pine belt extended from Virginia to Texas and covered an estimated 100,000 square miles of virgin forest.

The term "naval stores" was originated in the days of wooden sailing ships and included tar, pitch, rosin, turpentine, cordage, masts and yards. Although the term still persists, naval stores today consist chiefly of rosin and turpentine, but pine oil, tar, depentene, terpene hydrocarbons, and tall oil are also included.

In almost 350 years of history, there have been few fundamental changes in the original method of production of gum naval stores. Today, however, less than 5 per cent of the total naval stores production is used in the shipbuilding industry. Table 1 lists the industrial users of turpentine and rosin in the 1950 naval stores year.

Table 1 - Turpentine and Rosin Uses, 1949-1950*

Industry	Turpentine, barrels(1)	Rosin, drums(2)
Abattoirs	0	368
Adhesives and plastics	149	20,096
Asphaltic products	117	885
Automobiles and wagons	50	19
Chemicals and pharmaceuticals	75,623	329,867
Ester gum and synthetic resins	15,720	199,862
Foundries and foundry supplies	564	3,332
Furniture	112	4
Insecticides and disinfectants	35	2,314
Linoleum and floor covering	0	30,994
Oils and greases	17	15,683
Paint, varnish and lacquer	12,141	93,360
Paper and paper size	0	365,740
Printing ink	156	5,492
Railroads and shipyards	4,438	7,980
Rubber	168	9,611
Shoe polish and shoe materials	3,039	4,382
Soap	0	74,041
Other industries	113	3,387
	112,442	1,167,417

* Annual period from April 1 through March 31.
(1) Barrels of 50 gallons.
(2) Drums of 520 pounds net weight.

Taken from 1949-1950 Annual Naval Stores Report, U. S. Dept. of Agriculture.

As early as 1900 the Federal Government urged that new sources of naval stores be found because it was feared that lumbering and poor manufacturing would soon seriously affect production in this country.

The original method of production was destructive distillation of the heartwood in crude earthen kilns. Later, the method of "tapping" live trees was started and this method, with refinements, is still used to gather the crude gum.

Today, four methods are used in the United States in the production of naval stores:

(a) Steam and solvent extraction of pine stumps or deadwood, which produces wood rosin and wood turpentine, pine oil and other terpenes.

(b) Distillation of crude gum or oleoresin obtained by tapping live trees. Products are gum rosin and gum turpentine.

(c) The recovery and refining of black liquor soap from sulfate pulping of pine wood, which results in sulfate wood turpentine and tall oil.

(d) Destructive distillation of pine wood in retorts, which results in destructively distilled turpentine, pine tar, and pitch.

As late as 1920, it was generally believed that within another ten years, gum production would be at an end. This belief initiated the further development of the wood naval stores industry. However, improved forestry practices have resulted in an abundant second growth of longleaf and slash pine trees, that have practically assured the supply of gum naval stores indefinitely.

The wood naval stores industry had its beginning in 1907 when Homer T. Yaryan, a native of Indiana, although more than 60 years of age, became interested in the value of dead pine wood and stumps. He conceived the idea of grinding the wood and extracting the essential oils with a solvent. After laboratory experiments indicated that this could be done, a plant was built at Gulfport, Mississippi, in 1909, embodying the fundamental principles of this industry. This plant, in its first 12-month season, produced more than 14,000 barrels of rosin.

In 1911 the Yaryan organization built another plant at Brunswick, Georgia, with a capacity of 300 tons of wood per day. During the 1912-1913 season the two Yaryan plants produced 9,800 barrels of rosin and 15,000 barrels of turpentine.

Due to a drop in the price of turpentine, both plants closed in 1913, but with a more favorable market in 1914, the plants were reopened. It is of interest to note that wood naval stores plants are still in operation at these locations.

The original method of extraction used in the wood industry, and still used in some plants, was to steam the turpentine from the chips and extract heavier oils and rosin with gasoline. The gasoline used at that time could not be separated from the turpentine by fractional distillation because of the wide boiling range. Today all wood naval stores plants, except Hercules, use a narrow-range gasoline. The Hercules plants at Brunswick, Georgia, and Hattiesburg, Mississippi, use coal tar hydrocarbons - largely benzene and toluene. This type

of solvent will give a higher yield, at a sacrifice of color, and is used primarily when the rosin is to be subsequently refined.

Primarily because of engineering development and research, the wood naval stores industry has flourished and at present there are fourteen plants operated by nine companies in five Southern States, the most recent plant being constructed in Louisiana in 1947. The Hercules plant at Hattiesburg began operations in the fall of 1923.

These fourteen plants process more than two million tons of dead pine wood per year, and annually clear more than one million acres of cutover southern pine forests. The supply of stumps is estimated to last an additional 15 to 30 years.

Research currently being conducted on the use of ponderosa pine wood may eventually lead to wood naval stores plants being constructed in the Pacific Northwest.

7. Composition and Uses of Naval Stores Products

Steam distilled wood turpentine consists chiefly of terpene hydrocarbons, of which about 80 per cent is alpha-pinene. Beta-pinene is present in small amounts. The only other dicyclic hydrocarbon found is camphene, also only in small amounts. The remainder of the hydrocarbon portion consists of monocyclic hydrocarbons, notably dipentene limonene, terpinene, and terpinolene. The nonhydrocarbon portion, less than 2 per cent of the turpentine, consists of secondary and tertiary terpene alcohols, aldehydes, phenols, phenolic ethers, and oxidation and polymerization products. Of the secondary alcohols,

both borneol and fenchyl are present. Of the teritary alcohols, alpha-terpineol is found and constitutes a substantial part of the total alcohols. Small amounts of benzaldehyde, furfural, cineol, soberol, camphor, cymene and methyl chavicol are also present.

Turpentine finds its greatest use in paints, varnishes, and lacquers. Other uses are in chemicals and pharmaceuticals, shoe polish and shoe materials, and in the synthesis of camphor.

Pine oil is distinctly a product of the heartwood and stumps of pine trees, and is not found in the exudations from living trees. Its composition depends upon the method of production. Steam-distilled pine oil contains terpene hydrocarbons, borneol and fenchyl alcohols, and terpene ethers, present in amounts varying from 5 to 10 per cent. Alpha-terpineol constitutes 50 to 60 per cent, other terpineols 15 to 25 per cent, and ketones and phenols 1 to 2 per cent. Pine oil also contains small amounts of vanillin, terpin hydrate, and anethol. Pine oil is believed to be formed in nature from the pinenes through oxidation and hydrolysis.

Pine oil is used as a frothing agent in the mining industry for the separation of metals by the flotation process. It is also used as a wetting agent in the textile industry, and in medicinal preparations because of its antiseptic properties.

Steam-distilled wood rosin consists of about 90 per cent acidic material. Of this acidic material, approximately 90 per cent is abietic acid and its isomers. The remaining acids are dihydroabietic and dehydroabietic acids. Besides acid constituents, rosin has about

10 per cent neutral material called resenes, a few of which have been identified and appear to be mixtures of compounds which to date have no outlet other than as a part of rosin.

Rosin is manufactured in many grades corresponding to color standards established by the United States Department of Agriculture, the paler grades generally being the more desirable. Rosins are graded according to a letter system of D, E, F, FF, G, H, I, K, M, N, WG, WW, and X, with D being the darkest and X being the lightest grade. The grade FF applies only to the rosin produced by the steam-distillation and solvent-extraction of pine stumps. Grades F to WG can be produced from FF wood rosin by further refining to remove the color bodies.

The uses of rosin depend on the physical properties of the various grades and modifications produced. Rosin finds extensive use in the paper, soap, paint, and varnish industries. These industries consume about two-thirds of all rosin produced in the United States. Rosin is becoming increasingly important in the manufacture of chemicals, pharmaceuticals, and in synthetic rubber and resins.

Dipentene, a constituent of crude wood turpentine, is used as a softener in rubber processing and reclaiming. Terpin hydrate, anethol, and other terpene chemicals find use in medicinal preparations, perfumes, and as special solvents.

8. Raw Materials

The principal raw materials are the stumps and lightwood of two southern pines - longleaf yellow pine (Pinus palustris Miller) and slash pine (Pinus caribaea Morelet).

The stumps were left in the ground from lumbering operations and the original method of handling the live pines in gum naval stores production, which frequently killed the tree or so weakened it that it fell during high winds, common in the coastal states.

The wood structure is largely heartwood with comparatively thin sapwood. The heartwood is saturated with terpenes and resins, making it almost impervious to attack by insects, fungi, or rot. The sapwood, or less durable portion, with small resin content, has largely been removed by decay. Both stumps and lightwood, which is dead topwood, that have been in or on the ground for many years, are preferred. Stumps that have not "seasoned" are difficult to reduce to the proper size for extraction. The average composition of seasoned pine stumps includes 22 per cent moisture, 5 per cent volatile oils, 22 per cent rosin, and 51 per cent ligneous and cellulose materials.

In addition to the stump wood, other raw materials used include benzene, furfural, and gasoline solvents, in the primary operations and various chemicals and compounds used in specialized or secondary plant operations. The most common of the chemicals and compounds used in secondary operations, and which may be present in the process wastes, are:

Sulfuric Acid
Glycerine
Pentaerythritol
Maleic Acid
Tall Oil
Ammonia
Ethylene Glycol
Di and Tri Ethylene Glycol
Methanol
Acetic Acid
Rosin Oils
Rosin Soaps
Terpenes
Soda Ash
Lime
Dowtherm (Diphenyl, Diphenyloxide).

9. Production

The average daily production based on 800 tons processed was reported as follows:

Rosin (including "Belro" and "Vinsol")	320,000 pounds
Turpentine (including Pinene)	4,000 gallons
Pine Oil	2,400 gallons
"Solvenol" (including Dipentene)	2,400 gallons

Production data from secondary units - largely batch processes - were not reported on a daily basis, but were given for the month of February, 1951, as follows:

"731" Resin	2-1/2 million pounds
"Poly-Pale" Resin	3-1/2 million pounds
Synthetic Resins	3 million pounds

The product yields depend, to a great extent, on the resin content of the wood processed, which varies with the locality from which the stumps were gathered.

10. Operations

The Hercules plant at Hattiesburg operates 24 hours a day (3 shifts, 7:00 AM, 3:00 PM, 11:00 PM) seven days a week. The plant is shut down one or two weeks each year for major repairs and overhaul.

11. Employees

The plant employs approximately 1,000 persons, exclusive of those engaged in stump removing and hauling activities.

12. Water Supply

Water for use in plant processes is drawn from two sources: Bowie River, which supplies the bulk of the process water; and a deep

well, which supplies boiler feed water and some process water. Total pumpage averaged 4,800 gallons per minute. Used condenser water is returned to a cooling basin for re-use. Recirculation through these spray ponds was estimated to be 2,500 to 3,000 gallons per minute. Spray pond loss due to evaporation was given as 300 gallons per minute, and miscellaneous uses amount to 200 gallons per minute.

13. Waste Disposal

All process wastes from the plant, except the stump washer, are discharged into a main ditch running through the plant proper. The main ditch discharges directly into the Bowie River. The water used in stump washing operations is used condenser water and when not needed for washing stumps is discharged directly into the main ditch. The effluent from washing the stumps discharges into Hartfield Creek which empties into the Bowie River about one mile above the main plant effluent.

14. Sanitary Wastes

Sanitary wastes are discharged to the Hattiesburg sewerage system. No evidence of sanitary wastes being discharged in the process waste sewers was found.

DISCUSSION OF PROCESSES

The Hattiesburg plant of the Hercules Powder Company is a wood naval stores plant in which all products are derived directly or indirectly from pine stumps. Crude products obtained, by steam and solvent extraction, from the stumps, are refined and modified in various ways to produce a large variety of products. The general operation of the plant is outlined below.

15. Stump Transportation and Storage

Pine stumps are dislodged from the ground by "pushdozers" or dynamite, and delivered to the plant by trucks or rail. About 160 stump trucks now operate between woods and plant. A 30 or 40 mile haul is considered the economic limit for trucking. Approximately 65 per cent of all stumps are brought in by rail from Louisiana, Alabama, and Mississippi.

After delivery of the stumps to the plant, the wood is unloaded by an electric crane with an "orange peel" bucket. The crane operates on a 200-foot runway over a storage pit holding 700 to 800 tons of wood. Storage space is also available for stockpiling 6 months to 1 years supply of stumps for use when field activities are halted due to unfavorable weather.

16. Stump Washing

The stumps are moved by chain conveyor through a water spray where dirt, sand, and loose or rotted wood is removed. This wash water is passed over a coarse screen for recovery of large pieces of wood.

The water, carrying silt and small wood fibers, flows by gravity to Hartfield Creek.

17. Size Reduction

The clean stumps are fed directly to grinding equipment consisting of diamond type "hogs" which reduce the stumps to chips 2 to 3 inches in length by 1/4 to 1/2 inches in diameter. These chips are then screened and fed to Jeffrey shredders which deliver wood chips 1/2 to 1-1/2 inches in length and 1/8 inch diameter. This size is considered optimum, allowing the maximum rate of extraction with a minimum difficulty in handling. The chips are moved by chain and belt conveyors to the fresh chip storage bins to await processing.

The stump washer and shredding equipment is operated 16 hours out of 24, from 10:00 AM to 6:00 PM, and 10:00 PM to 6:00 AM.

18. Extraction

Fresh chips are moved by belt conveyors to the top of the extractor house where they can be loaded in any of the extractors. The chips are introduced through the top of the extractors, packed in tightly and all openings bolted tight.

The extraction is carried out in a battery of 15 pressure vessels, each holding 15 tons of chips, under elevated temperature and pressure. The extractors are worked in cycles, the fresh solvent being introduced into the extractor containing the most exhausted chips, and the loaded solvent being passed through the freshest chips.

At the conclusion of the cycle, the chips are steamed to remove solvent and liquid pine products.

The spent chips are removed manually from a door near the bottom of the extractor and moved by belt conveyor to spent chip storage until needed for fuel to produce steam for process use and electric power generation.

19. Drop Solution Tanks

The crude solution, known as drop solution, is passed through storage tanks which permit greater flexibility of operations. A small amount of water separates from the liquid pine products and is drained daily from these tanks to the main ditch.

20. Preliminary Refining

The crude solution is pumped to the still house to be fractionated in bubble-cap type fractionating columns to obtain the desired cuts. Three fractions are removed in this process: recovered solvent, crude oils, and crude rosin. The recovered solvent is returned to the system to be re-used. About one per cent liquid pine products remain in the system solvent. The crude oils and rosin pass on to further refining processes.

All waste water from preliminary refining is collected and reprocessed to recover solvent before being discharged to the main ditch.

21. Crude Oil Refining

The crude oils obtained from the preliminary refining process are further refined by fractional distillation into a variety of blended liquid products. There are three general classes of oils present: bicyclic terpenes, monocyclic terpenes, and terpene alcohols. Separation of these oils is carried out only to the extent necessary to produce commercially marketable products.

There is no continuous flow of waste water, other than surface condenser water, from crude oil refining. Waste waters discharged periodically, but not sampled, include crude turpentine wash water and still wash water. These wastes are discharged to the main ditch.

22. Rosin Refining

The crude rosin, free from oils, is further processed by selective solvent refining. The crude rosin is dark-colored and contains about 20 per cent gasoline-insoluble material. The rosin is refined by treating, while hot, with gasoline. This gasoline solution is further treated with furfuraldehyde which selectively dissolves the color bodies. The solution separates into two layers; the lower or furfural layer contains the darker-colored material, largely gasoline-insoluble, and is marketed as "Vinsol" resin. The gasoline layer contains the lighter-colored rosin which when further treated with furfural in refining towers, yields the "Pexite" grades of rosin equivalent to U. S. Standards F to WG. The residual solution from the towers contains "Belro," which is a special intermediate grade peculiar to furfural refining of the crude rosin.

The various rosins are recovered from solution by suitable evaporators. Although Pexite rosins may be produced in many grades; the types produced at Hattiesburg are, except for special reasons, K, M, and N grades. At present all of the rosin is used in secondary units to produce more valuable resins or to be compounded into other products.

All waste water is recovered and processed to remove gasoline and furfuraldehyde before being discharged to the main ditch.

A flow diagram of the primary process is given in figure 1.

23. Secondary Refining

The preliminary refining processes outlined above are basic to all other operations. All secondary refining carried out at the plant uses the above products as raw materials.

24. Poly-Pale Process

Poly-Pale resin is produced by treating Pexite rosin with sulfuric acid in benzol solution, under very closely controlled conditions of temperature, concentration, and contact. Under these conditions, about one-third of the rosin acids present unites to form polymers. The remaining two-thirds of the rosin constituents are practically unchanged. The resulting resin is superior to rosin in making ester gums of commerce. It has a high melting point, 20°C. higher than average rosins, is more resistant to oxidation, has no tendency to crystallize, and in solution has a higher viscosity than the rosin from which it was made. It is light in color, WG to X when graded according to U.S.D.A. standards.

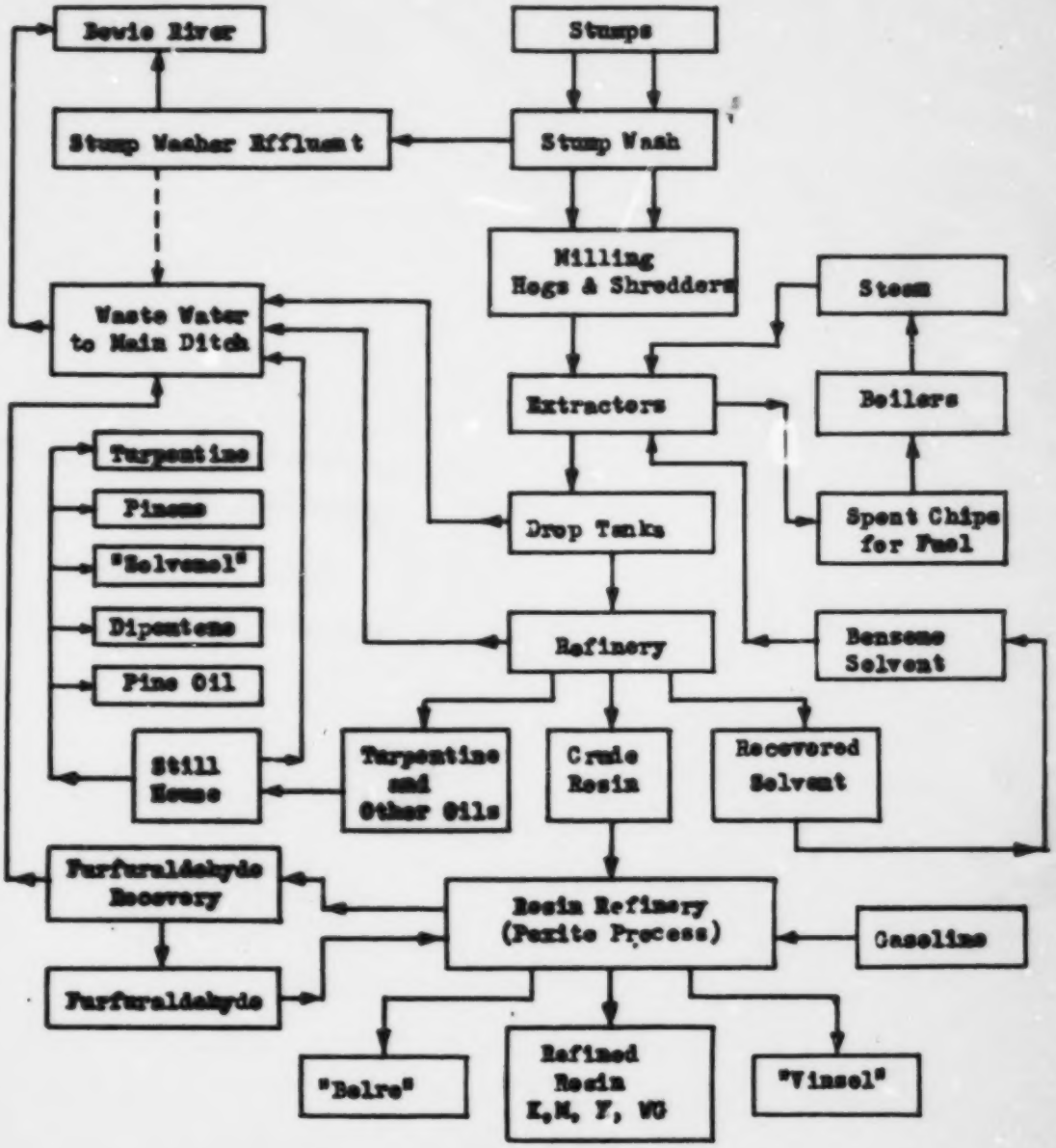

FIGURE 1 Diagram of Primary Process

The residual resin from the polymerization process is very dark in color, high in polymers, and has a high melting point. This resin is marketed as "Melhi."

In addition to surface condenser water, two process wastes were sampled from this operation: barometric condenser water from a sulfuric acid concentrator, and water processed for recovery of solvent. The solvent recovery unit is cleaned daily, using 50 pounds of soda ash. Lime is added to the waste water to raise the pH before being discharged to the main ditch.

A flow diagram of the Poly-Pale process is given in figure 2.

25. 731 Plant

731 resin is produced by treating Pexite rosin with heat in the presence of a catalyst. The resulting resin contains dehydroabietic acid, tetrahydroabietic acid, and some dihydroabietic acid. This reaction is generally referred to as disproportionation or dehydrogenation. The principal use of this resin is in preparation of "Dresinate," a rosin soap used in large volume in the manufacture of synthetic rubber.

Soap or size is also made in this unit using dark Belro resin or light Pexite rosins or combinations of both by mixing with alkali in large batch kettles.

The major waste water from this process is barometric condenser water from the vacuum system on the rosin distillation unit.

A flow diagram of the 731 plant process is given in figure 3.

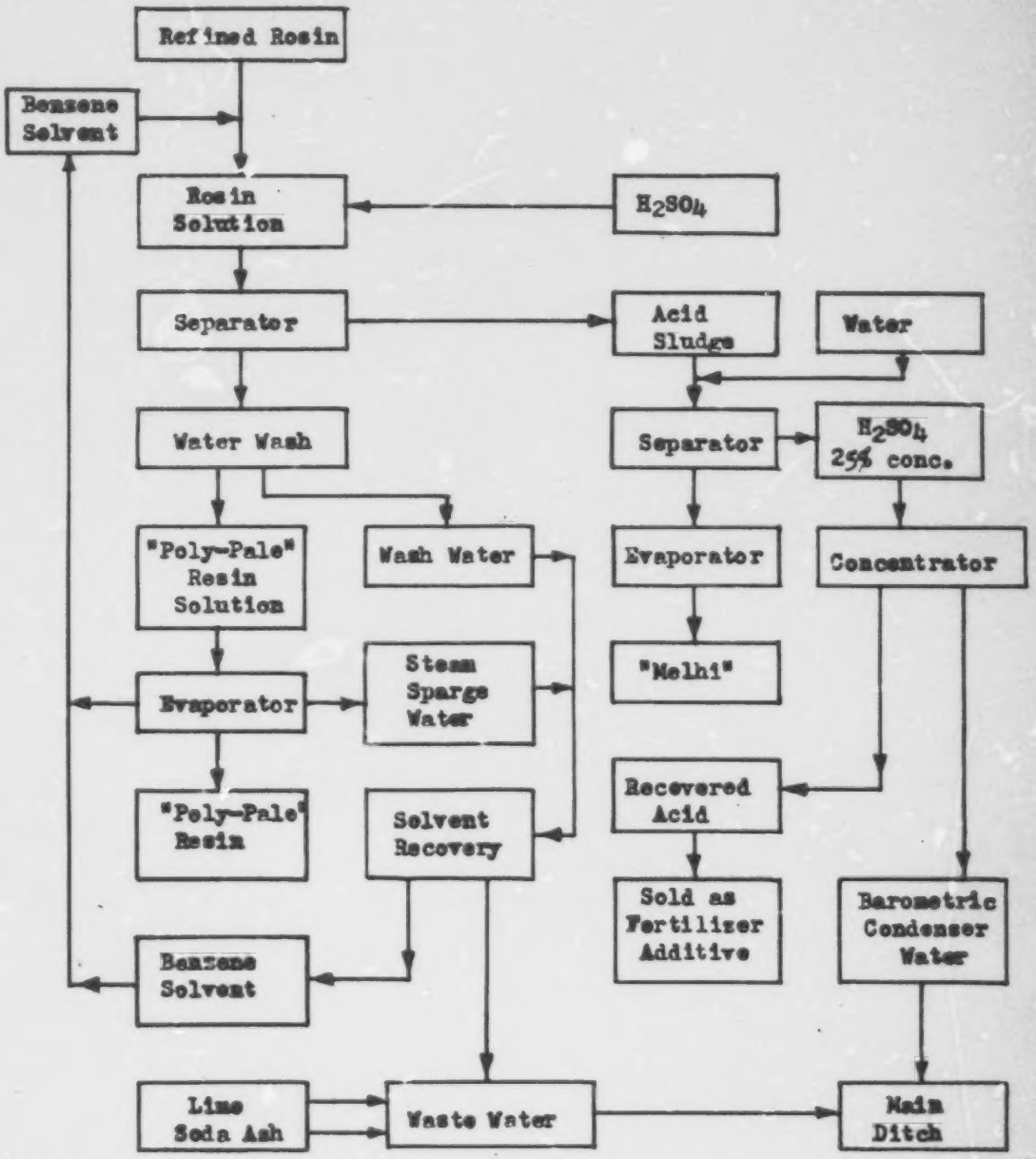

FIGURE 2 Diagram of Poly-Pale Plant Process

HERCULES NAVAL STORES SURVEY
HATTIESBURG, MISSISSIPPI
UNITED STATES PUBLIC HEALTH SERVICE

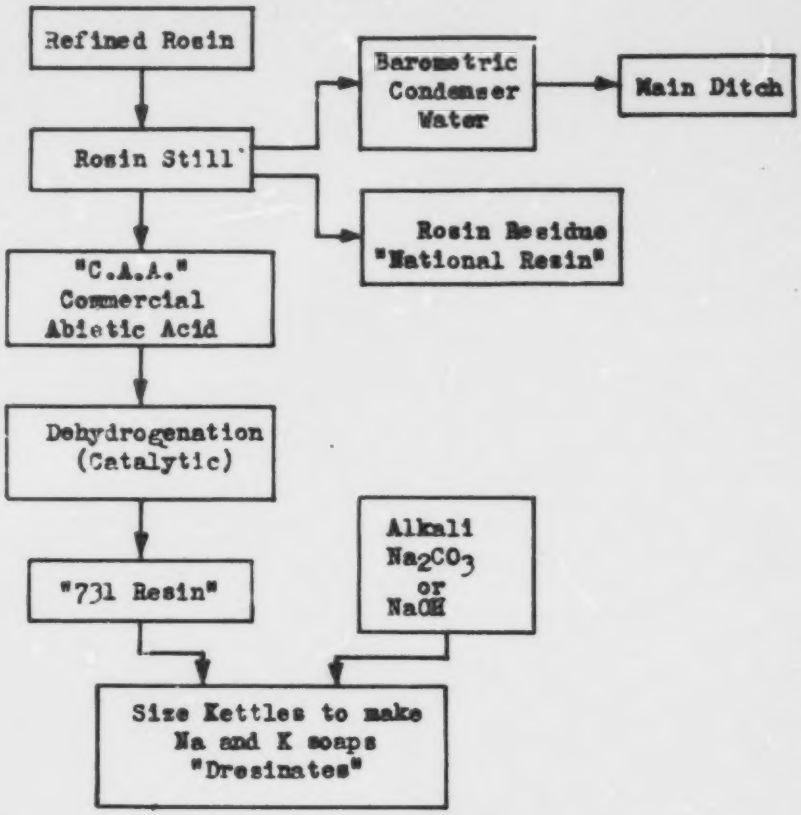

FIGURE 3 Diagram of "731" Plant (Size and Dresinate) Process

26. Hydrogenation Plant

Hydrogenated resin is produced by treating Pexite rosin with hydrogen in the presence of a finely divided nickel catalyst under high pressure. The hydrogen is produced at the plant by the cracking of natural gas. The resulting resin is known as "Staybelite" and is a glass-clear pale yellow resin very resistant to oxidation and discoloration on exposure to air or ultraviolet light. The waste liquids from this plant include small volumes of rosin oils and alkaline water used in processing the catalyst. These wastes are discharged directly to the main ditch.

27. Synthetics Plant

A variety of synthetic resins, ester guns, esters, and modified esters are produced in the synthetics plant by reacting Pexite, Staybelite, Poly-Pale, or 731 resins with various chemicals and compounds.

The synthetic resins are made in heated kettles by reacting the various resins with glycerine, Pentaerythritol, maleic anhydride, ethylene glycol, di and tri ethylene glycol, ammonia, hydrogen, acetic acid, and other materials.

Other processes carried out in this unit include production of the methyl ester of tall oil, and production of rosin amine and rosin amine acetate from rosin nitrile.

Waste water from the synthetics plant includes barometric and surface condenser water, and quench water used in knocking out fumes

produced by the batch processes. These wastes discharge to the main ditch.

A flow diagram of the synthetics plant process is given in figure 4.

28. Isomerization

In the isomerization process, liquid pine oil is treated with solid NaOH to convert methyl chavicol to anethol. Heat is applied to the solution for six hours, allowed to cool, and the NaOH removed by a series of two washes. Anethol is an aromatic liquid with a fragrant odor and taste of anise or licorice and is used in medicines and perfumes.

The liquid wastes from this process consist of the washings of the treated pine oil. The isomerization process is scheduled in the plant on an average of four times per week. The waste water averages 400 gallons per wash and is discharged to the main ditch.

29. Tank Car Washing

As a general service to the various plant units, returned tank cars are cleaned before being put back in use. The cars are normally steamed before being treated with a hot aqueous solution of sodium carbonate. The tank cars as a rule carry Hercules products, so the wastes from cleaning will contain products already described. Whenever possible, the cars are used for the same product, which minimizes cleaning. The number of cars cleaned varies from one or two per week to a maximum of two per day.

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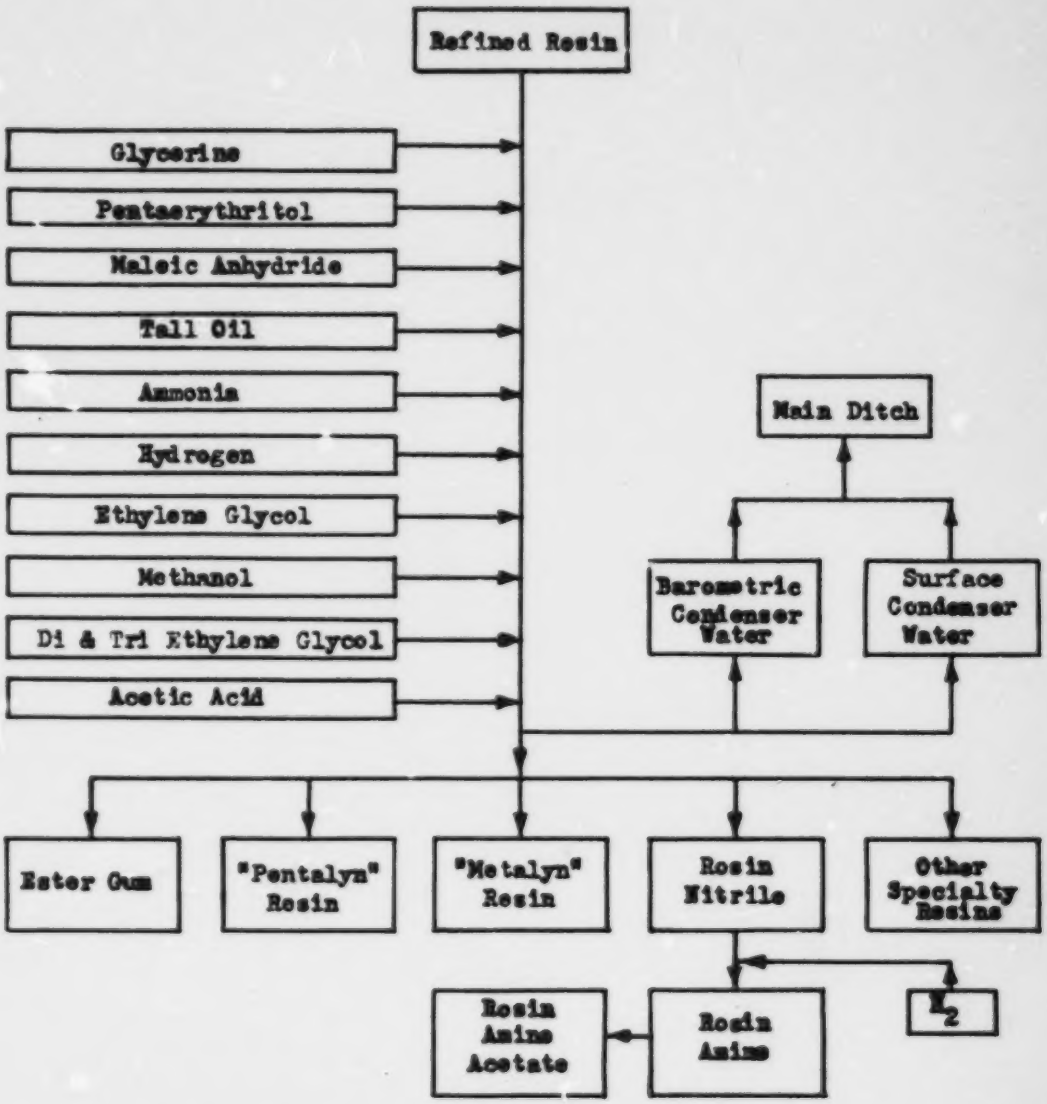

FIGURE 4 Diagram of Synthetics Plant Process

The cars are cleaned over two pits, both draining to the main ditch. The liquid wastes average about 500 gallons per wash.

30. Toxaphene Plant

The toxaphene plant is a new unit which was not in operation during the period of the survey. Toxaphene is a chlorinated camphene used as an insecticide by cotton farmers to control the boll weevil. The waste waters from this unit should contain little or no organic material. It is predicted that the pH of the waste water will be on the acid side, and equipment for neutralization with lime has been installed.

SURVEY PROCEDURES

31. Sample Collection

During the survey, samples were collected by plant personnel from the following sources: stump washer, 731 plant, solvent recovery, synthetics plant, furfuraldehyde recovery, Poly-Pale plant, drop tanks, tank car washing, and main plant effluent. These samples were collected at one-hour intervals between 8:00 AM and 3:00 PM and composited, except the drop tanks and tank car washings which were collected as the tanks or cars were being drained.

In addition, special samples were taken of the synthetics plant and main plant effluent for phenol determinations. These samples were collected at the same time as the regular samples. In an

attempt to obtain a plant waste water balance, samples were also collected from the hydrogenation plant, spray pond overflow, isomerization wash water, 7th Street drainage ditch, still house, Poly-Pale ammonia condenser, 731 plant miscellaneous flow, and plant influent. These samples were grab samples collected at the times indicated on the data summary sheets.

32. Flow Measurements

Flow measurements at the Poly-Pale, 731, and synthetics plants and the main plant effluent were made by means of sharp-crested, rectangular weirs installed by plant engineers for the duration of the survey. The heads on the weirs were checked hourly by the sampler by reference to an established point, and the flows were computed from Francis's formula:

$$Q = 3.33 L H^{3/2}$$

where, Q - flow in cubic feet per second

L - length of weir in feet

and H - head on weir in feet

Correction for end contractions of the Poly-Pale plant and main ditch weirs were computed from Francis's formula for end contractions:

$$L = L' - 0.1 N H$$

where, L - corrected length of weir in feet

L' - measured length of weir in feet

N - number of end contractions

and H - head on weir in feet.

Miscellaneous flow from the 731 plant, which included floor washings, accidental spills, oil tank vents, and some surface runoff, was measured by means of a 90° V-notch weir. The head on the weir was noted when the sample was collected and the flow computed from King's formula:

$$Q = 2.52\ H^{2.47}$$, using the same symbol notation as above.

Waste water measurements from the still house, furfuraldehyde recovery, isomerization wash, tank car wash, Poly-Pale ammonia condenser, drop tanks, solvent recovery, spray pond, acid concentrator, and plant influent were determined by plant personnel by various methods.

These methods included heat balances, pumpage records, volumetric measurements, and flow recording devices. Wherever possible these measurements were checked at the time of the survey.

Flow from the hydrogenation plant was determined volumetrically by accurately timing the filling of a container of known volume. Flow in the 7th Street ditch and stump washer effluent was determined by using a Price current meter. The stump washer effluent was also checked by noting the increase in the main ditch when the flow was diverted that way.

33. Laboratory Procedures

All or part of the following determinations were made on the samples collected:

B.O.D., 5 days at 20°C.
Oxygen Consumed
pH

Alkalinity or Acidity
Color
Ether-extractable material
Phenols
Total Solids, Volatile and Fixed
Suspended Solids, Volatile and Fixed

In addition, determinations for terpin hydrate, dissolved oils, and furfural content were made on special samples by the Hercules laboratory.

The procedures followed in making the above analyses are, in general, as given in "Standard Methods for the Examination of Water and Sewage," 9th Edition. Modifications of the procedures used for the determination of phenols, ether-extractable material, and oxygen consumed are given in the appendix of this report.

EXPLANATION OF LABORATORY DETERMINATIONS

34. Biochemical Oxygen Demand (B.O.D.)

The biochemical oxygen demand of sewage, sewage effluents, polluted waters, or industrial wastes, is the oxygen required during stabilization of the decomposable organic matter by aerobic bacterial action. Since this is a phenomenon into which both time and temperature enter, a standard set of conditions (5 days at 20°C.) is universally used for the test. For normal domestic sewage and some industrial wastes, approximately 68 per cent of the oxygen requirement of the carbonaceous material present is satisfied during the standard test. Because of the variable carbon-nitrogen ratio of

industrial wastes, however, the rate of oxygen demand of such wastes may not follow the typical oxidation curve for domestic sewage. Results are expressed in parts per million (p.p.m.).

35. Oxygen Consumed

The oxygen consumed test is a measure of the chemical oxidant required to oxidize the organic matter present in a sample under particular standard conditions. The test is of value in estimating the strength of certain industrial wastes, and in conjunction with the determination of the B.O.D. It can be used where it would be advantageous to obtain results indicative of oxygen demand in less time than the biochemical oxygen demand test requires. Results are expressed as parts per million oxygen.

36. pH

The symbol pH denotes the negative logarithm (to the base 10) of the hydrogen ion concentration of a solution, this symbol being a universal one in chemistry. The pH value indicates the relative acidity or alkalinity of a water or waste, being lower with higher degrees of acidity. It is a measure of intensity rather than quantity. All pH determinations, during this survey, were made using a Backman electrometric pH meter, Model G.

37. Alkalinity

Alkalinity of a water or waste represents the carbonates, bicarbonates, hydroxides, and occasionally the borates, silicates

and phosphates of the alkali and alkali earth metals. By use of the phenolphthalein and methyl orange end points, the amount of each type of alkalinity present can be determined. Methyl orange alkalinity is usually referred to as total alkalinity. Calcium and magnesium account for most of the alkalinity present in a sample. Results are expressed as parts per million calcium carbonate ($CaCO_3$).

38. Acidity

Acidity of a water or waste is usually caused by the presence of uncombined carbon dioxide, mineral acids and salts of strong acids and weak bases. By using phenolphthalein and methyl orange indicators, the amounts of each type of acidity present can be determined. Phenolphthalein acidity is usually referred to as total acidity, while methyl orange acidity is referred to as mineral acidity. Results are expressed as parts per million calcium carbonate ($CaCO_3$).

39. Color

True color of a water or waste is due to the presence of coloring matter in solution. Apparent color is due to color in suspension in in the water and can be removed by sedimentation or filtration. True color is usually caused by extracts of decomposing vegetable matter or, in the case of industrial wastes, by chemicals or compounds from processing various raw materials. Color is expressed in parts per million and is measured by comparing the water sample with prepared standards of potassium chloroplatinate. Apparent color determinations were not made on any samples during this survey.

40. Ether-Extractable Material

Ether-extractable materials include such substances as oils, greases, and tars. The presence of these pollutants renders water difficult and sometimes impractical to treat, either for industrial or domestic use. In addition, these substances make beaches unsightly, coat watercraft, and are a hazard to fish and waterfowl. Results of laboratory determinations are expressed in parts per million.

41. Phenols

The laboratory determinations for phenols indicate the gross phenol content including cresols and higher phenols. These substances are found in variable proportions and amounts in certain industrial wastes.

Phenols in quantities as low as 0.002 to 0.005 parts per million in the presence of chlorine will produce chlorophenol (medicinal) tastes and odors in water. A concentration of 0.1 part per million of phenol alone imparts to water the characteristic taste and odor of phenol. In fish, phenol produces paralysis of the neuromuscular mechanism and hemolyzes the blood.

42. Solids

The determination of solids includes those for total and suspended solids as well as their volatile constituents. Total solids consist of all the solids present in a liquid. The term includes both suspended and dissolved solids. Suspended solids are in suspension or

float on the surface of water, sewage or other liquid, and are removed by laboratory filtration. Dissolved solids are found by subtracting suspended solids from total solids. Volatile solids are those that are lost upon ignition of total solids (giving total volatile solids) or suspended solids (giving suspended volatile solids). Fixed solids are determined by subtracting total volatile solids from total solids (giving total fixed solids) or suspended volatile solids from total suspended solids (giving fixed suspended solids). Volatile solids represent approximately the amount of organic matter, and fixed solids the inorganic matter in a water or liquid waste.

Suspended solids in streams cause water to be turbid and unsightly and may be deposited in streams, interfering with navigation,and injury to the habitat of fish.

43. Population Equivalent

Normal domestic sewages, relatively free from industrial wastes, possess characteristics which are reasonably well defined, and it is, therefore, possible to compare them on a common basis. Industrial wastes are influenced by the processes from which they originate and the characteristics are not readily defined by the same standards. The "Population Equivalent" value, therefore, provides a basis on which industrial wastes may be compared to domestic sewages. The coefficient,

0.17 pounds of 5-day, 20°C., B.O.D. per capita per day,

used in comparing domestic sewages, has been used in converting the data to a population equivalent basis.

DISCUSSION OF RESULTS

Although the plant operated continuously, the plant processes were not necessarily the same during each 24-hour period. This is due, in part, to the many batch processes that are not scheduled on a regular daily basis. Variations are also partly due to general plant clean-up, tank draining, and tank car washing, that are normally attended to on the daylight shifts. The amounts of products, by-products, and wastes are affected by the quality of the stumps processed. The strengths of wastes produced cannot be correlated with production because of indeterminate storage periods throughout the process.

In cases where process water already contains some impurities, the quality of the water leaving the plant and the various units must be compared to the incoming water in order to obtain a fair estimate of the concentrations of the wastes discharged. This procedure has been followed in analyzing the data presented in this report. Table 2 gives the average concentration and quantities of the plant influent.

Tables 3, 4, and 5 show the important findings of the survey. Table 3 shows the average concentration of significant constituents of the wastes sampled, while table 4 shows the average daily quantities of these constituents. Table 5 shows the percentage of the constituents contributed by each component of the main ditch.

44. Discharge

The average daily plant discharge amounted to 5.332 million gallons, of which the stump washer contributed 0.432 and the main ditch 4.90 million

Table 2 - Average Concentrations and Quantities of Significant Constituents of Plant Influent (Bowie River Water)

Estimated Flow		
	GPM	3,500
	MGD	5.04
B.O.D. at 20°C.		
5-day	ppm	1.7
	lbs/day	73
pH		6.8
Total Alkalinity		
	ppm	10
Oxygen Consumed		
	ppm	15
	lbs/day	630
Total Solids		
Total		
	ppm	132
	lbs/day	5,540
Volatile		
	ppm	38
	lbs/day	1,600
Fixed		
	ppm	94
	lbs/day	3,940
Suspended Solids		
Total		
	ppm	46
	lbs/day	1,930
Volatile		
	ppm	17
	lbs/day	715
Fixed		
	ppm	29
	lbs/day	1,220

Table 3 - Average Concentrations of Significant Constituents

Source	Discharge M.G.D.	B.O.D. 5-day 20°C.	Oxygen Consumed	Ether Extractable Material	Alkalinity T-Total P-Phenolphthalein	Acidity T-Total M-Mineral	Total Solids			Suspended Solids		
							Total	Volatile	Fixed	Total	Volatile	Fixed
		Parts per million										
Stump washer	0.432	186	5730			51 T	11100	5290	5810	9140	4080	5060
Main plant effluent	4.900	246	531	62.8	100 T		426	168	258	134	68	66
Hydrogenation plant	0.000036	382000	958000	467000			410000	410000	0	26500	26500	0
731 plant(miscellaneous)	0.012	38100	79000		3520 T 380 P		14600	11400	3100	3350	3325	25
Isomerization wash water												
1st wash	0.0004	20100	18300		116000 T 90000 P		128000	37600	90000	75	33	42
2nd wash	0.0004	9450	13400		6380 T 3450 P							
Furfuraldehyde recovery	0.0096	10700	14500	21		190 T 120 M	151	115	36	7.5	6.1	1.4
Drop tanks	0.000104	4110	10200	660		840 T 200 M	5370	5020	350	595	571	24
Solvent recovery	0.115	2080	2790	30		300 T 170 M	150	111	39	16	12	4
Still house oil trap effl.	0.0098	1370	1380	10.9			79	46	33	44	8	36
Synthetics plant	0.600	271	830	312	560 T 370 P		415	252	163	130	114	16
731 Plant(dehydrog'n)	0.332	256	592	79	14 T		150	79	71	41	27	14
7th St.drainage ditch	0.252	164	274				165	69	96	39	25	14
Poly-Pale solv.recovery	0.243	76	430	20.8		880 T 750 M	1530	960	570	28	13	15
Acid concentrator	0.086	17	29			114 T 96 M						
Poly-Pale ammonia condenser	1.250	2.0										
Spray pond overflow	0.432	1.3					104	29	72	50	14	36

Table 4 - Average Daily Quantities of Significant Constituents

Source	Discharge M.G.	B.O.D. 5-day 20°C. lbs.	Oxygen Consumed lbs.	Ether Extractable Material lbs.	Total Solids			Suspended Solids		
					Pounds					
					Total	Volatile	Fixed	Total	Volatile	Fixed
Stump washer	0.432	668	20,600		40,000	19,000	21,000	32,900	14,700	18,200
Main plant effluent	4.900	10,000	21,700	2,560	17,430	6,890	10,540	5,480	2,790	2,690
731 Plant (miscellaneous)	0.012	3,880	7,900		1,480	1,170	310	341	339	2
Solvent recovery	0.115	2,000	2,670	29	143	107	36	15	11	4
Synthetics plant	0.600	1,390	4,150	1,600	2,120	1,280	840	667	586	81
Furfuraldehyde recovery	0.0096	856	1,160	1.7	12	9	3	0.6	0.5	0.1
731 Plant (dehydrogenation)	0.332	703	1,640	220	411	217	194	112	73	39
Total still house waste	0.936	483			819	257	562	499	94	405
7th Street Drainage Ditch	0.252	345	575		347	144	203	82	52	30
Poly-Pale Solvent Recovery	0.243	139	870	52	2,990	1,930	1,060	57	27	30
Hydrogenation Plant	0.000036	115	288	140	123	123	0	8	8	0
Isomerization Wash Water										
First wash	0.0004	67	61		425	125	300	0.25	0.11	0.14
Second wash	0.0004	32	43							
Poly-Pale Ammonia Condenser	1.250	21								
Acid Concentrator	0.086	12.2	21							
Spray Pond Overflow	0.432	4.7			3.6	1.0	2.6	1.8	0.5	1.3
Drop Tanks	0.000104	3.6	8.9	0.6	4.6	4.3	0.3	0.52	0.50	0.02

Table 5 - Percentage Contributions of Significant Constituents of Main Plant Effluent

Source	Discharge	B.O.D. 5-day 20°C.	Oxygen Consumed	Ether Extractable Material	Total Solids			Suspended Solids		
					Total	Volatile	Fixed	Total	Volatile	Fixed
Main plant effluent	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
731 Plant (Miscellaneous)	0.2	38.8	36.4		8.5	17.0	2.9	6.2	12.2	0.07
Solvent recovery	2.3	20.0	12.3	1.1	0.8	1.6	0.3	0.3	0.4	0.2
Synthetics plant	12.2	13.9	19.1	62.5	12.2	18.6	8.0	12.2	21.0	3.0
Furfuraldehyde recovery	0.2	8.6	5.3	0.07	0.07	0.1	0.03	0.01	0.02	0.004
731 Plant (Dehydrogenation)	6.8	7.0	7.6	8.5	2.4	3.1	1.8	2.0	2.6	1.3
Total still house waste	19.1	4.8			4.7	3.7	5.3	9.1	3.4	15.0
7th Street drainage ditch	5.1	3.4	2.6		2.0	2.0	1.9	1.5	1.9	1.1
Poly-Pale solvent recovery	5.0	1.4	4.0	2.0	17.2	28.0	10.0	1.0	1.0	1.1
Hydrogenation plant	0.000007	1.2	1.3	5.5	0.7	1.8	0.0	0.1	0.3	0.0
Isomerization wash water										
1st wash	0.08	0.6	0.3		2.4	1.8	2.8	0.005	0.004	0.005
2nd wash	0.08	0.3	0.2							
Poly-Pale ammonia condenser	25.5	0.2								
Acid concentrator	1.8	0.1	0.1							
Spray pond overflow	8.8	0.05			0.02	0.01	0.02	0.03	0.02	0.05
Drop tanks	0.002	0.04	0.04	0.002	0.03	0.06	0.003	0.01	0.02	0.001
Plant influent		0.7	3.0		31.0	22.5	36.4	34.3	24.9	44.0
Total	87.2	101.0	92.2	79.7	82.0	100.2	69.4	66.8	67.8	65.8
Unaccounted for	12.8		7.8	20.3	18.0		30.6	33.2	32.2	34.2

gallons. The stump washer effluent remained remarkably constant, while the main plant effluent varied considerably. The maximum flow in the main ditch exceeded the average by 7.5 per cent, and the minimum was 10.7 per cent less than the average. This difference in flow was due to variations in plant operations.

45. Stump Washer Effluent

The stump washer effluent proved to be a serious offender, contributing daily to the Bowie River an average of 40,000 pounds of total solids. This was more than twice the amount contributed by the main plant effluent.

Although the daily poundage of 5-day B.O.D. was small, as compared with the main plant effluent, the volatile solids were extremely high, indicating that more organic material was present in the waste than was detected by the B.O.D. determination. Caution must be taken in using B.O.D. figures for this waste, as it contains wood fibers that are not easily broken down by microbiological action. In addition, this waste contains tannins, in solution, which are powerful reducing agents.

The dark brown color of this waste was also due mainly to tannin extracts from the bark of the stumps. All tannins are polyhydroxylic phenols and have bactericidal properties that not only affect the B.O.D. determination but also the biology of the receiving stream.

46. Main Plant Effluent

The main plant effluent contains all process waste water except the stump washer effluent. This waste varied considerably both in quantity and concentration. These variations can be attributed to the following process wastes that make up the main plant effluent.

731 Plant - Two wastes were sampled from this unit, one containing the process waste and the other made up of floor washings, accidental spills, oil tank vents, and some process water. Considerable variations in concentrations were noted and are due to batch method of manufacture. These wastes averaged 45.8 per cent of the B.O.D. of the main plant effluent.

Solvent Recovery - The primary process solvent recovery unit contributed 20 per cent of the main plant B.O.D. and remained fairly constant during the period of the survey.

Synthetics Plant - The synthetics plant waste varied considerably due to the numerous products processed. This waste accounted for the largest percentage of ether-extractable material and suspended solids.

Poly-Pale Solvent Recovery - Although this unit was responsible for less than 2 per cent of the total B.O.D., the total volatile solids amounted to 28 per cent. Daily variations in concentrations were due mainly to the changing amounts of surface condenser water discharged with the solvent recovery waste.

Seventh Street Drainage Ditch - The values reported for this sample point are not all chargeable to plant operations. In addition to pilot plant and laboratory wastes, this ditch contained surface runoff

water. It was impracticable to determine the exact quantities and concentrations of all contributing sources. Approximately one-half the dry-weather flow is attributed to surface runoff and one-half to plant wastes. Almost all B.O.D. and solids, however, can be charged to plant wastes.

Hydrogenation Plant - Liquid wastes from this unit remained constant during the period of the survey. Since the volume is so small, 36 gallons per day, and 100 per cent volatile, it is possible that this waste can be disposed of by incineration, or other methods.

Tank Car Wash - This was was extremely variable in concentration, depending upon the type of car washed. The B.O.D. quantities had a range of 0.1 pound to 1030 pounds per car wash.

Other Components - Other components sampled but which proved to be of minor significance included drop tanks, acid concentrator, furfuraldehyde recovery, total still house waste, isomerization wash water, and poly-pale ammonia condenser. These individual wastes were quite variable in concentration, but averaged more than 15 per cent of the main plant B.O.D. The results obtained from these sample points were also used in preparing a daily waste water balance.

Table 6 shows the average daily waste water balance. Population equivalents of the various wastes are given in table 7.

Data summary sheets for all sample points are included in the appendix of this report.

Table 6 - Average Daily Waste Water Balance

Source	Discharge, million gallons	B.O.D. 5-day 20°C. lbs.	Oxygen Consumed lbs.	Ether Extractable Material lbs.	Total Solids			Suspended Solids		
					Total lbs.	Volatile lbs.	Fixed lbs.	Total lbs.	Volatile lbs.	Fixed lbs.
Plant influent										
Bowie River water	5.040									
Well water	1.440									
Total	6.480	75	630		5,540	1,600	3,940	1,930	715	1,220
Total plant effluent*	5.332	10,700	42,300	2,560	57,400	25,900	31,500	38,400	17,500	20,900
Net chargeable to plant	5.332	10,625	41,670	2,560	51,860	24,300	27,560	36,470	16,780	19,680
Total plant constituents*	4.700	10,720	40,000	2,040	48,900	24,400	24,500	34,700	15,900	18,800
Unaccounted for	0.632	+ 100	1,670	520	2,960	+ 100	3,060	1,770	880	880
Per cent unaccounted for	12.9		4.0	20.3	5.7		11.1	4.9	5.2	4.5

* Includes stump washer effluent.

\+ Indicates an amount in excess of that chargeable to plant operations.

Table 7 - Population Equivalents of Process Wastes

Source	Population Equivalent
Stump Washer	3,900
Main Plant Effluent	59,000
731 Plant (Miscellaneous)	23,000
Solvent Recovery	12,000
Synthetics Plant	8,200
Furfuraldehyde Recovery	5,000
731 Plant (Dehydrogenation)	4,100
Total Still House Waste	2,800
7th Street Drainage Ditch	2,000
Poly-Pale Solvent Recovery	820
Hydrogenation Plant	680
Isomerization (Both Washes)	580
Poly-Pale Ammonia Condenser	120
Acid Concentrator	70
Drop Tanks	20

Based on 0.17 pounds B.O.D. per capita per day.

SUMMARY

Based on data and supporting information presented in this report, certain points may be summarized as follows.

47. Quantity of Liquid Wastes Discharged

The average daily discharge amounted to 5.332 million gallons, distributed as follows:

Source	Average Flow	Per Cent of Total	Low Flow	High Flow
	Million Gallons per Day			
Stump washer effluent	0.432	(100)	0.432	0.432
Main Plant effluent	4.90	(100)	4.38	5.27
731 Plant (miscellaneous)	0.012	0.2	0.012	0.012
Solvent recovery	0.115	2.3	0.115	0.115
Synthetics plant	0.600	12.2	0.526	0.641
Furfuraldehyde recovery	0.0096	0.2	0.0096	0.0096
731 Plant (Dehydrogenation)	0.332	6.8	0.302	0.410
Total still house waste	0.936	19.1	0.936	0.936
7th Street drainage ditch	0.252	5.1	0.252	0.252
Poly-Pale solvent recovery	0.243	5.0	0.108	0.352
Poly-Pale ammonia condenser	1.250	25.5	1.250	1.250
Acid concentrator	0.086	1.8	0.086	0.086
Spray pond overflow	0.432	8.8	0.432	0.432
Hydrogenation plant	0.000036	0.000007	0.000036	0.000036
Isomerization wash water	0.0008	0.16	0.0008	0.0008
Drop tanks	0.000104	0.002	0.000104	0.000104
Other Sources*	0.632	12.8		

* Includes wash water, condenser water, and miscellaneous wastes not sampled.

48. Quantity of Significant Constituents

Total pounds of constituents discharged from the plant daily are as follows:

Constituent	Total Pounds in Effluents*
5-day B.C.D.	10,700
Oxygen consumed	6,260
Ether-extractable material	2,560
Total solids	57,400
Total volatile solids	25,900
Total fixed solids	31,500
Total suspended solids	38,400
Suspended volatile solids	17,500
Suspended fixed solids	20,900

* Includes both stump washer and main plant effluents.

49. Unit Values

The unit values for wastes and constituents resulting from the processing of one ton of stumps are as follows:

	Stump-Washing Wastes	Process Wastes
Waste water, gallons	540	6,120
5-day B.O.D., pounds	0.84	12.5
Oxygen consumed, pounds	25.8	27.1
Ether-extractable material, pounds		3.2
Total solids, pounds	50.0	21.8
Population equivalent, persons	4.9	73.8

50. Average Yield

The average yield of rosin and liquid pine products per ton of stumps processed is as follows:

Rosin, all grades	400 pounds
Turpentine, including Pinene	5 gallons
Pine Oil	3 gallons
Solvenol, including Dipentene	3 gallons

SPECIAL STUDIES

Throughout the survey, special studies were conducted on some of the plant units. These studies were undertaken in an attempt to discover other factors that influence the characteristics of wood naval stores wastes. The Hercules laboratory by request of Mr. Frank Gardner, research chemist, made several analytical determinations on waste samples submitted by the Public Health Service field unit.

51. Terpin Hydrate

A study of the reduction of B.O.D. and oxygen consumed quantities by removing terpin hydrate from the wastes was conducted during the period February 28 - March 2, 1951. Duplicate samples were collected from the solvent recovery and furfuraldehyde recovery units. One sample was analyzed by the Public Health Service mobile laboratory for significant constituents, and the other sample was submitted to the Hercules laboratory for the quantitative determination and removal of terpin hydrate. The sample with the terpin hydrate removed was then further analyzed by the mobile laboratory for B.O.D. and oxygen consumed concentrations.

The results of these studies show that the average terpin hydrate content of the solvent recovery was 700 parts per million, and of the furfuraldehyde recovery unit 4,830 parts per million. The B.O.D. of both wastes was reduced 62 per cent by removal of terpin hydrate. The oxygen consumed was reduced 66 per cent in the solvent recovery waste and 62 per cent in the furfuraldehyde recovery waste.

Terpin hydrate is not ordinarily recovered from these units unless the market price is favorable. From the standpoint of stream improvement, it is desirable that this compound be removed from the liquid wastes.

52. Dissolved Oils

In a special study of the still house waste, duplicate samples were collected from the still house oil trap effluent on three consecutive days, April 2-4, 1951.

One set of samples was analyzed by the mobile laboratory and the other set submitted to the Hercules laboratory for the quantitative determination and removal of dissolved oils. The samples with the oils removed were further analyzed by the mobile laboratory for B.O.D. and oxygen consumed concentrations.

Results of this study show that the average dissolved oil concentration was 330 parts per million. The B.O.D. was reduced 67 per cent and oxygen consumed 71 per cent by removing the dissolved oils. These oils are not usually recovered from the still house waste.

53. Phenols

In an attempt to determine if phenols were present in the waste, special samples were collected from the synthetics plant, isomerization wash water, and main plant effluent. The main plant effluent contained an average of 14.2 pounds of phenols per day. The isomerization wash water and synthetics plant effluent, which are components of the main plant effluent, contained an average of 7.7 pounds per day. This left 6.5 pounds per day that were not found.

Hays, Drake, and Pratt,[1] in a study of disproportionated rosin soap (731 plant dresinate), found phenolic materials in the unrefined soap. Their findings show 0.11 per cent phenolic materials of weakly acidic character and 0.17 per cent of slightly stronger phenols, based on total unrefined disproportionated rosin. The exact amount of phenols being discharged with the process waste has not been determined. Since phenols and phenolic ethers are present in liquid pine products, it is possible that phenols could also be present in other liquid plant wastes. This was not determined during the survey.

54. Furfural

A study to determine the presence of furfural was undertaken because this compound is known to be a principal constituent of many industrial wastes. Furfural is used in this plant as a selective solvent in the refining of crude rosin.

Ten samples were collected from the furfuraldehyde recovery unit on separate days and analyzed by the Hercules laboratory for furfural content. Nine of the ten samples analyzed had less than 10 parts per million, and one sample contained 90 parts per million furfural. This waste was reported by plant personnel to be the only process water containing this compound. Although furfural is very persistent and will remain in a waste for a relatively long period of time, the dilution factor will reduce the concentration to less than 0.2 parts per million in the main plant effluent.

55. Main Plant Effluent Studies

Since most of the floor washing, tank draining, and general cleanup was confined to the day shift and consequently would give a more concentrated waste during this period, a special study was made of the main plant effluent during all three work shifts.

Hourly samples were collected and composited. Results of three 24-hour composite samples indicate that the flow remained constant throughout all shifts. B.O.D., ether-extractable material, and solid content varied considerably. A tabulated summary, based on percentage of constituents discharged on each shift, follows:

Sample Period	B.O.D. at 20°C. 5-day. % of 24-Hour Total	Ether-Extractable Material % of 24 Hour Total	Total Solids. Per Cent of 24-Hour Total			Suspended Solids. Per Cent of 24-Hour Total		
			Total	Volatile	Fixed	Total	Volatile	Fixed
8 AM - 3 PM	48.4	37.0	42.2	47.0	39.6	45.3	37.2	56.0
4 PM - 11 PM	25.8	31.5	28.9	26.5	30.2	27.4	31.4	22.0
12 Mid.- 7 AM	25.8	31.5	28.9	26.5	30.2	27.4	31.4	22.0

Samples were composited on an 8-hour and 16-hour basis; no differentiation was made for the second and third shifts, as operations during these periods remained fairly constant.

Throughout the survey, grab samples of the main plant effluent were collected to determine what variations occur in concentration due to tank draining, tank car washing, still washing, and accidental spills,

which are all low-volume, high-concentration, wastes. Samples were taken without regard to time, plant operations, or other factors that might affect the waste.

Results of 11 grab samples show a mean B.O.D. concentration of 397 parts per million as compared to 246 parts per million concentration reported on regular composited samples. The B.O.D. concentrations varied from 189 to 1295 parts per million.

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ACKNOWLEDGMENT

It is desired to express appreciation to Mr. L. C. Hall, Plant Manager of the Hattiesburg Naval Stores Division, and to Mr. Bruce W. Dickerson, Sanitary Engineer, of the Hercules Powder Company, for their co-operation and assistance extended during the conduct of this survey. Special mention must be made of Mr. Frank Gardner, Research Chemist, who gave generously of his time and counsel.

Virgil A. Minch
Jr. Asst. San. Eng. (R)

Albert L. Platz
Jr. Asst. San. Eng.

APPENDIX

DEFINITIONS OF TERMS PERTAINING TO NAVAL STORES AND RELATED PRODUCTS**

Abietic Acid, Commercial Grade - A mixture of rosin acids sometimes separated from rosin (or tall oil) in either crystalline or vitreous transparent form, and in which true abietic acid is a typical constituent, along with its isomers.

Acid Number - The numbers of milligrams of KOH necessary to neutralize one gram of rosin.

Alpha-pinene - See Pinene.

Alpha-terpineol - A tertiary alcohol, $C_{10}H_{20}O$, found in liquid pine products.

Anethol - Methylallylphenol, $C_{10}H_{12}O$, a fragrant solid of liquid found in small quantities in pine oil.

Belro* - Intermediate grade of rosin peculiar to furfural refining of crude rosin.

Beta-pinene - See Pinene.

Camphene - A terpene hydrocarbon found in pine wood. ($C_{10}H_{16}$).

Colophony - A term denoting medium and high grades of rosin.

Dehydrogenated Rosin - See "731" Rosin, and Rosin, Modified.

Dipentene - A monocyclic terpene hydrocarbon, the optically inactive form of limonene, present in the higher boiling fractions of several kinds of turpentine. Commercial dipentene usually also contains other terpenes and related compounds in varying amounts.

Disproportionated Rosin - See "731" Resin, and Rosin, Modified.

Dresinate* - A rosin soap made from "731" resin, used in large quantities in the manufacture of synthetic rubber.

Essential Oil - Any one of a class of odoriferous, volatile liquids, insoluble in water. Some contain dissolved rosin and are known as oleoresins.

Hydrogenated Rosin - See Rosin, Modified.

Melhi* - Residual resin from polymerization of various grades of rosin.

Naval Stores - Chemically reactive oils, resins, tars, and pitches derived from the oleoresin contained in, exuded by, or extracted from trees chiefly of the pine species, or from the wood of such trees.

Niggre - The dark, oxidized resinous material present in and extracted from wood along with rosin, and subsequently removed therefrom by washing or selective extraction.

Oil of Turpentine - The pharmaceutical name for spirits of turpentine which conforms to the requirements of the U. S. Pharmacopoeia.

Oleoresin - Pine gum, the nonaqueous secretion of resin acids dissolved in a terpene hydrocarbon oil which is:

(1) Produced or exuded from the intercellular resin ducts of a living tree.
(2) Accumulated, together with oxidation products, in the dead wood of weathered limbs and stumps.

Pine Gum - See oleoresin.

Pinene - A bicyclic terpene hydrocarbon, the principal constituent of all turpentine, and existing there in two isomeric forms, alpha- and beta-pinene. The latter is found in appreciable quantity only in gum spirits and sulfate wood turpentine. Pinene not otherwise described usually means alpha-pinene.

Pine Oil - A colorless to amber-colored volatile oil with characteristic pinaceous odor, consisting principally of isomeric tertiary and secondary cyclic terpene alcohols, with variable amounts of terpene hydrocarbons, ethers, ketones, phenols and phenolic ethers, the amount and character of which depend on the source and method of manufacture. The three commercial kinds of pine oil are:

(1) Steam-distilled Pine Oil, obtained from pine wood by steam distillation or by solvent extraction followed by such distillation.
(2) Destructively-distilled Pine Oil, obtained from the lighter distillate from the destructive distillation (carbonization) of pine wood.
(3) Synthetic Pine Oil, obtained by chemical hydration of terpene hydrocarbons to form the terpene alcohols, or by dehydration of terpin hydrate.

Pine Needle Oil - An essential oil of typical fragrance obtained by steam distillation of the needles of certain species of pine or other coniferous trees.

Pine Pitch - The dark-colored to black solidified material, somewhat pliant and tenacious, obtained by distilling off practically all the volatile oil from a retort pine tar; the genuine contains no added free rosin.

Pitch, Navy - A pitch obtained by melting rosin with pine tar, with or without rosin distillation residues.

Pexite* Rosin - Trade name of various furfural refined rosins.

Poly-Pale* Resin - A high melting resin produced by treating rosin with sulfuric acid in a solvent solution.

Resenes - As applied to naval stores, those constituents of rosin which cannot be saponified with alcohol alkali, but which contain carbon, hydrogen, and oxygen in the molecule.

Resinates, Metallic - Rosin in which all or part of the rosin acids have been chemically treated with those metals which give soap or salts which are water-insoluble. Limed rosin, zinc-treated rosin, and the resinates of lead, cobalt, copper, and manganese, are of the greatest industrial importance.

Rosin - A specific kind of natural resin obtained as a vitreous water-insoluble material from pine oleoresin by removal of the volatile oils. It consists primarily of tricyclic monocarboxylic acids having the general empirical formula $C_{20}H_{30}O_2$, with small quantities of compounds saponifiable only with boiling alcohol potassium or sodium hydroxide, and some unsaponifiable matter. The two general classifications or kinds of rosin in commerce are:

(1) Gum Rosin, obtained from the oleoresin collected from living trees.
(2) Wood Rosin, obtained from the oleoresin contained in dead wood, such as stumps and knots.

Rosin, Limed - See Resinates, Metallic.

Rosin, Modified - Rosin that has been treated with heat or catalysts, or both, with or without added chemical substances, so as to cause substantial change in the structure of the rosin acids, as isomerization, hydrogenation, dehydrogenation, or polymerization, without substantial effect on the carboxyl group. The following are types of modified rosin:

(1) Disproportionated (dehydrogenated) Rosin - Rosin which has been subjected to chemical or physical treatment, or both, so as to cause substantial simultaneous hydrogenation and dehydrogenation of the rosin acids to form their hydrogenated and dehydrogenated counterparts.
(2) Heat-Treated Rosin - Rosin in which a reduction of acid number and a positive shift in optical rotation has been brought about by controlled heat treatment only, in order to improve its suitability for specific uses.
(3) Hydrogenated Rosin - Rosin which has been treated with hydrogen under conditions which will cause a partial or complete saturation of the rosin acids present, best indicated by a drop in the refractive index. Commercial hydrogenated rosin is usually only partially saturated.

(4) Polymerized Rosin - Rosin which has been treated by chemical or physical means, or both, in a manner so as to cause a union of a part of the rosin acids to form polymers of greater molecular weight, to such an extent that the molecular weight of such rosin will be measurably greater than that of the original rosin.

Rosin Acids - The acidic components of rosin isomeric with the acids present in oleoresin, with empirical formula $C_{20}H_{30}O_2$, and derived therefrom when the oleoresin is converted to rosin by the usual commercial processes.

Rosin Oil - The relatively viscous, oily portion of the condensate obtained when rosin is subjected to dry destructive distillation; also used to describe specially compounded oils having a rosin oil base.

Rosin Spirits - The relatively light, volatile portion of the condensate obtained in the first stages when rosin is subjected to dry destructive distillation.

Rosin Standard (Grade) - Any combination or assembly of colored glasses prepared and issued by U. S. Department of Agriculture for use in classifying rosin as to grade. Standards for the following grades have been established by or under authority of the Federal Naval Stores Act: Grades X, WW, WG, N, M, K, I, H, G, F, E, D, and FF. FF grade is used only for wood rosin. Rosin darker in color than the standards for D or FF is graded B.

731* Resin - A resin produced by treating various grades of rosin with heat in the presence of a catalyst. The principal use is in the preparation of Dresinate*, a rosin soap.

Solvenol* - A mixture of monocyclic and bicyclic terpene hydrocarbons used as a softener in rubber processing.

Staybelite* - Trade name of a hydrogenated rosin.

Synthetic Resins - A variety of modified compounds, made in most cases by treating refined or semirefined rosins with different chemicals or compounds.

Tall Oil - A supersaturated solution of rosin acids related to abietic acid, and of fatty acids related to oleic acid, together with nonacidic bodies, which is obtained by acidifying black liquor skimmings of the alkaline pulp industry.

Terpene (hydrocarbon) - An unsaturated organic compound having the empirical formula $C_{10}H_{16}$, occurring in most essential oils and oleoresins of plants; further classified as monocyclic (dipentene), dicyclic (pinene), or acyclic (myrcene), according to the molecular structure.

Terpene Alcohol - An alcohol directly related to or derived from a terpene hydrocarbon; the following are common examples: terpineol (tertiary cyclic), borneol (secondary cyclic), geraniol (primary, acyclic), linalool (tertiary, acyclic).

Terpin Hydrate - Dipentene glycol, $C_{10}H_{20}O_2.H_2O$, obtained by oxidation of turpentine and used medicinally or in the manufacture of synthetic pine oil.

Turpentine, (Spirits of) - The volatile oil consisting primarily of a number of terpene hydrocarbons of the general formula $C_{10}H_{16}$, obtained by distilling the oleoresin exuded by or contained in the wood of certain species of pine trees. Four kinds of turpentine are now recognized:

(1) Gum Turpentine or Gum Spirits of Turpentine, made from the gum (oleoresin) collected from living trees.
(2) Steam-Distilled Wood Turpentine, obtained from the oleoresin within the wood by steam distillation of the wood direct or of an extract therefrom.
(3) Sulfate Wood Turpentine, recovered during the conversion of wood to paper pulp by the sulfate process.
(4) Destructively-Distilled Wood Turpentine, obtained by fractionation of certain oils recovered by condensing the vapors formed during the destructive distillation of pine wood.

Turpentine, Oil of - The pharmaceutical name for spirits of turpentine which conforms to the requirements of the U. S. Pharmacopoeia.

Turpentine, Sulfite - This term is not in good usage, because the volatile oil recovered in the conversion of wood to pulp by the sulfite process consists chiefly of cymene ($C_{10}H_{14}$) rather than pinene and other terpenes.

Turpentine Substitute - This term is not recommended, because its use to describe a paint thinner of mineral oil origin is illegal under the Federal Naval Stores Act.

Vinsol* - A low grade rosin, soluble in furfural, but not in gasoline, used in foundry cores and as an admixture in Portland cement.

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* Registered trade names of commercial products manufactured by Hercules Powder Company.

** Taken in part from American Standards for Testing Materials, part 4, Naval Stores, Paint, Wood, Adhesives, Paper, and Shipping Containers, page 572, 1949.

The Industrial Waste Survey

The approach to each industrial waste survey follows the same general plan, the program being modified as necessary to conform with requirements of a specific plant.

State Authority

Each project is scheduled through and with approval of the State stream control agency. State technical personnel occasionally collaborate on these studies.

Objective

The industrial waste survey is designed to determine the total pollution load from an industry and to indicate the source, quantity, concentration, and characteristics of each major individual waste. Constituent balances are computed when feasible.

Plant Operations

These studies are conducted at a time when plant operations are normal, in so far as this is possible. In correlating plant wastes with stream conditions, attempt is made to study the stream over a range of discharge with emphasis being placed on critical low flow.

Organization

The Public Health Service field staff assigned to industrial waste studies is normally composed of:

Experienced engineer	1
Assistant engineer	1
Experienced chemist	1
Assistant chemist	1

A biologist may be assigned to the field unit where bioassays or biological stream studies are indicated. The Environmental Health Center technical staff is available for consultation on technical questions arising from these projects and, under certain conditions, may undertake short-time special studies having direct bearing on the problem.

Company Representation

The industrial representative who collaborates in organization, planning, conduct of survey, and review of data, is usually from the research department but may be from any department within the company charged with the responsibility of studying the plant wastes. Knowledge of the plant and process is a prime requisite for this individual.

Mobile Laboratory Requirements

This field unit is currently equipped with two trailer laboratories. Each trailer laboratory should be allowed ground space of approximately 11' x 30' and at a location where electricity, water, and sewers are available. The equipment will require a 3-wire (110-220 V), 60 cycle, alternating current circuit and about 11 KW.

Flow Measurements

Choice of methods employed in measuring the volumes of wastes will depend on the local situation. In general, that method should be employed, in each case, which promises to give the most accurate results under the operating condition. Volumetric measurements are frequently possible. Continuous flows can be measured by any one of several methods with weirs, equipped with liquid level recorders; Parshall flumes being suitable for open channel flow. It is expected that the industry will provide personnel for installation of necessary flow-measuring equipment.

Sampling

The objective in all sampling is to obtain a truly representative sample of each waste examined. Each sample is a composite consisting of portions taken at frequent regular intervals during each sampling period of the survey. If the rate of flow varies appreciably, the sample portions are proportioned to the flow. The sampling day may vary between 8 hours and 24 hours, depending on the uniformity of operations. Normally the sampling program can be so organized that one sampler for each shift can collect all samples and record necessary data. When operations are uniform from hour to hour, it is occasionally possible to confine sampling to the day shift. It is expected that industry will provide personnel for sampling.

Process

It is imperative that sufficient information be made available, pertaining to process and plant operation, to make possible correct interpretation of the analytical data obtained from the survey. Cognizance is taken of the fact that each manufacturing plant may employ

special operation procedures, developed by that organization, which might be termed secret processes. It is realized that some of these developments may have been patented whereas others may have been considered of greater value without resorting to protection through patent procedure. Our engineers wish to become familiar, only in a general way, with various manufacturing processes in order that the major waste sources will be recognized. Our representatives hold in strictest confidence any process or operating information, obtained during the survey, so requested by the industry.

Determinations

The general sanitary criteria frequently are not well adapted to characterizing an industrial waste. Decision as to those determinations to be made is based on stream objectives and the parameters which best define the pollutional characteristics of the waste. Determinations to be made should be jointly agreed on by the industrial representative and the engineer in charge of the field unit, with the collaboration of the chemist who will perform the analytical work.

Analytical Data Review

Following completion of the field work and analysis of the data, the results are reviewed with the industrial technical staff prior to preparation of the survey report. This joint review of the data is considered advisable even though the U. S. P. H. S. field staff and the plant's technical personnel may work closely throughout the investigation.

Period of Study

The length of time required to conduct an industrial waste survey will depend upon the magnitude of the problem. In the larger plants, it is desirable to make a separate study of each major department. The survey in each department should extend through ten operating days, or longer if necessary, to give sufficient accurate data for averaging.

Report

The Public Health Service prepares a final report on the project and provides the industry with copies, these copies being forwarded through the State stream control agency. These reports are not considered official record until they have been reviewed and approved by the industry.

January 16, 1951

To: Record

From: Chief, Industrial Wastes Section

Subject: Hercules Powder Co., Inc., Hattiesburg, Mississippi, Industrial Waste Studies - Conference.

1. Date of Conference

January 11, 1951.

2. Personnel Participating

Following is a list of men participating in this conference, grouped by organization represented:

Hercules Powder Co., Inc.
L. C. Hall, Manager
R. J. Byrkit, Chief Chemist
Frank Gardner, Research Chemist
Bruce W. Dickerson, Sanitary Engineer

Mississippi State Board of Health
H.A.Kroeze, Director, Division of Sanitary Engineering
C. M. Ledbetter, Sanitary Engineer

U. S. Public Health Service
Virgil A. Minch, Junior Assistant Sanitary Engineer (R)
Hayse H. Black, Senior Sanitary Engineer.

3. Purpose

The meeting was scheduled by Mr. Kroeze for the purpose of discussing process wastes created by operations of Hercules Powder Company, Hattiesburg, Mississippi, plant,and their pollutional effect on Bowie River, a tributary of Leaf River. This project is a continuation of the study of liquid wastes from pine stump processing plants in the Gulf States.

GENERAL INFORMATION

The morning and early part of the afternoon was devoted to discussion of process and the wastes produced. Messrs. Gardner and Dickerson conducted a tour of the plant in the late afternoon and pointed out principal waste sources. This memorandum will serve as a record of this meeting.

4. Organization

This plant was originally constructed by Hercules Powder Company. It is understood that the plant was placed in operation in 1923.

The main offices of Hercules Powder Co., Naval Stores Department, is located at Wilmington, Delaware. The company maintains a branch Research and Development Laboratory at Hattiesburg, Mississippi, as well as a Control Laboratory. Mr. R. J. Byrkit directs the work of both laboratories.

5. Products

The principal products are refined rosins and liquid pine products. Two rosin products having different color are sold commercially as Vinsol and Belro. The liquid pine products include:

(a) Pine Oil (several grades)
(b) Turpentine
(c) Pinene
(d) Dipentene
(e) Solvenol.

A new department will be placed in operation later this spring for production of toxaphene. This chemical is used by cotton growers to control boll weevil.

6. Raw Materials

The basic raw material is the longleaf yellow pine stump. Solvents employed include benzene, gasoline and furfural. Other raw materials which may be present in the wastes are:

Methanol	Rosin Oils	Sulfuric Acid
Pentaerythritol	Rosin Soaps	Maleic Acid
Glycol	Terpenes	Anhydrous Ammonia
Ethylene Glycol	Caustic Soda	Dowtherm (Diphenyl,Diphenyloxide).
Glycerine	Soda Ash	
Tall Oil	Lime	

7. Capacity

This plant has a capacity for processing 300 tons of stumps per 24-hour day.

8. Operations

Extraction and refinery end of the plant operates continuously. Wood washing, shredders and hammer mills are operated on a split shift. The plant is normally shut down for maintenance work for one week during June or July.

The primary operations consist essentially of extraction, distillation, and refining to produce rosin and liquid pine products. Secondary operations are conducted for the purpose of producing further refined and additional products.

9. Water Supply

Bowie River is the principal source of process water. The water intake is located downstream from the stump washer discharge and roughly two miles above confluence of Leaf River.

Information regarding supplemental water supply should be determined during the survey period. This would include possible ground water from company wells as well as the Hattiesburg municipal water supply.

10. Sewerage

There are two sewer outlets from this plant, both discharging to Bowie River. One of these sewers carried stump washer water and the other was designated "main sewer."

Stump washer waste is normally discharged through a lagoon where much of the silt settles out. Currently, some experimental work is being conducted at the stump washer which permits occasional diversion of this waste water to the main sewer rather than the lagoon. Physical appearance of discharge from the main sewer indicated that some of the stump washer water was being diverted at the time of the plant inspection.

The main sewer discharge was estimated at 4,000 g.p.m. This discharge is composed of waste from both primary and secondary processes and normally includes all waste except the stump washer water.

Sanitary wastes are discharged to the Hattiesburg sewerage system. It was thought that one or two wash rooms in the plant might possibly discharge to the process sewers rather than the city system. These sewer connections will be checked in conjunction with the waste studies.

PRINCIPAL WASTE SOURCES

Consideration was given to principal departments and to those processes having direct bearing on the plant waste problem. Company representatives were familiar with the major individual wastes, although limited information was available on their volume and pollutional characteristics.

11. Stump Washer Water

Water is sprayed on the stumps as they are conveyed to the shredders. This water removes sand and silt adhering to the wood and also erodes some of the rotten wood from the stump surface. This wash water is passed over a coarse screen for removal of large pieces of wood. This stump washer water, carrying silt and small fiber particles, flows by gravity to the settling pond. Impoundment of this stump washer water was believed efficient in removal of silt, but some fiber escapes as indicated by its presence in the plant water supply taken from Bowie River downstream from the lagoon discharge.

12. Extractor House Waste

This waste would be expected to contain sugars, acetic acid, formic acid, benzene. Stump chips were reported to contain 20% to 25% moisture.

13. Still House Waste

This waste would be expected to contain small quantities of furfural, rosin soaps, and rosin oils. It was reported that this waste would be diluted with condenser water.

14. Rosin Refinery Waste

This waste was reported similar in character to that from the extractor house. Inaddition, it would be expected to contain some rosin oils and soda ash. The rosin refinery, still house, and extractor house are considered as primary operations, each with separate discharge to the main sewer.

15. Poly-Pale Operation

This process takes refined rosin and by polymerization produces two products. Sulfuric acid is used as a polymerization agent. Organic-acidic wastes are partially neutralized with soda ash.

This department has an acid concentrator from which there is a discharge approximately once a month, referred to as "still bottom." This acid concentrator operates under vacuum created by a barometric condenser supplemented by steam jet. There is possibly some entrainment of acid in the barometric condenser water.

16. Size and Dehydrogenation Plant

Discharge from this plant is primarily barometric condenser water and floor wash. This waste should be examined even though it is believed to be insignificant.

17. Synthetic Resin and Rosin Amine Plant

This waste contains barometric condenser water and floor wash. It is possible that the waste may contain some phenol. Discharge from this plant was through a flume at a relatively high velocity. Arrangements are to be made for measuring this flow, preferably by means of a weir.

18. Hydrogenation Plant

This department is included in this memorandum, for completeness. The process consists of hydrogenating rosin, and there are no organic wastes, according to the plant technical staff.

19. Tank Car Cleaning

A number of tank cars are cleaned daily. Some of these tank cars are company-owned and others are under contract. Occasionally cars are used that have contained materials other than the products for which they are to be used. This cleaning operation normally consists of steaming followed by a water wash. The volume and characteristics of these wastes may be quite variable.

SURVEY PROGRAM

Plant operations were believed to be sufficiently uniform to permit sampling for eight hours only out of the twenty-four. It was further stated that these wastes were relatively stable and no difficulty should be experienced in holding them overnight. If further study of these wastes confirm these opinions, it should be possible to sample during the day shift.

Some problems remain to be solved in connection with accurately measuring various discharges to be analyzed. One of the most important measurements is that for the main sewer which presently discharges over a broad-crested weir. More accurate measurements could be obtained by installation of a sharp-crested weir at this location. We have two Fries liquid level recorders for use in continuous recording of water levels back of weirs.

It may be possible to obtain accurate measurements of some of the major individual wastes by a series of volumetric determinations. Preliminary study of these hydraulic problems should indicate the preferred procedures to be used in obtaining these volumes. If the rate of flow varies more than plus or minus 10%, it is recommended that the samples be weighted.

The Hercules Powder Company technical staff was of the opinion that the Hattiesburg laboratory would not desire to split the samples. However, if portions of specific samples are desired for special or check analyses, sufficient quantities should be taken to make this possible. We have no objection to running any analyses in parallel by our laboratory and Hercules Powder Company laboratory. This procedure makes possible checking of techniques.

ANALYTICAL PROCEDURES

It was tentatively agreed to run the following sanitary analyses on these wastes. It will not be necessary to run all determinations on each waste, these decisions to be made by the Hercules Powder Company technical staff and the United States Public Health Service field unit, with knowledge of the waste being exmined:

5-day B.O.D.	pH
Oxygen Consumed	Color
Solids	Chlorides
Oils	Alkalinity
Phenol	Sulfides

It was pointed out that difficulty might be experienced with the colorimetric method for determination of pH. Accordingly, it is planned to determine pH potentiometrically.

STUDY OF RECEIVING STREAM

Company representatives specifically requested check analyses on Bowie River as a means of determining the pollutional effect of these plant wastes. U.S.G.S. records indicated flow in Bowie River to vary between 150 and 13,000 cubic feet per second. Normally, there is substantial flow in this stream during the month of February. If normal stream flow conditions prevail, it is possible that a stream study, as a part of the plant survey, might not give useful information. The preferred time for study of the stream would be during a period of critical low flow. It was tentatively agreed to make at least exploratory stream studies, the amount of work to be dependent on stream flow conditions.

PROJECT SCHEDULE

The field unit scheduled for the Hattiesburg, Mississippi, project should be available during the week of January 29 or the week of February 5. It is probable that the Hattiesburg plant study may extend through a period of four weeks. In the event stream studies give useful data, the project may well continue through the first two weeks of March. It was agreed to review analytical results at approximately the midpoint in this study. This procedure will afford opportunity to critically examine accumulated data and to review the remaining program. The exact time for this meeting with representatives of Hercules Powder Company, Mississippi State Board of Health, and U.S.P.H.S. will be determined at a later date.

SUMMARY

This memorandum is intended for use of the U.S.P.H.S. field unit in organizing a co-operative industrial waste study with Hercules Powder Company and the Mississippi State Board of Health. Inasmuch as this memorandum is based on fragmentary notes, information contained herein should be confirmed prior to inclusion in the survey report.

Hayse H. Black
Chief, Industrial Wastes Section
Survey and Investigation Branch

HHB/ecf

March 2, 1951

To: Record

From: Chief, Industrial Wastes Section

Subject: Hercules Powder Company, Inc., Hattiesburg, Mississippi, Industrial Waste Studies - Conference

1. Date of Conference

February 28, 1951.

2. Personnel Participating

Following is a list of men participating in this conference, grouped by organization represented:

Hercules Powder Co., Inc.
L. C. Hall, Manager
Frank Gardner, Research Chemist
Bruce W. Dickerson, Sanitary Engineer

Mississippi State Board of Health
H. A. Kroeze, Director, Division of Sanitary Engineering
C. M. Ledbetter, Sanitary Engineer

U. S. Public Health Service
Virgil A. Minch, Junior Assistant Sanitary Engineer (R)
Jesse Cohen, Chemist
Donald D. Gold, Senior Assistant Engineer
Albert L. Platz, Junior Assistant Sanitary Engineer
Morris B. Ettinger, Scientist
Hayse H. Black, Senior Sanitary Engineer

3. Purpose

This meeting was scheduled by Mr. Dickerson's February 15 letter and Mr. Kroeze's February 17, 1951, letters. The purpose of this meeting was to review available data from the co-operative industrial waste survey with Hercules Powder Company and to plan the remainder of this program. This memorandum records the principal considerations discussed at this conference.

4. Data

The field staff had tabulated analytical data for the period February 14 to February 23, 1951, inclusive. The more important waste data were averaged. These data were remarkably consistent and it was the consensus that irregularities could probably be explained by variations in process. Mr. Gardner offered to investigate plant operation records in an effort to explain major variations. There appeared to be a reasonably good correlation between 5-day B.O.D., oxygen consumed, and volatile solids.

5. Process

Four general flow diagrams had been prepared to facilitate interpretation of the waste data. These flow diagrams were designated: primary process, poly-pale plant, synthetic resin plant, and 731 plant (size and dresinate). Mr. Gardner will prepare a brief general statement covering the operations of each major department for use in the report and as an aid in the interpretation of the waste data.

6. Sampling

Samples to date have been composites made up of hourly portions collected during the day shift. It was pointed out that most of the floor washing was done on the day shift and that other operations confined to this shift would probably give a more concentrated waste during this 8-hour period than during the other two shifts. Accordingly it was decided to sample all points hourly 24 hours a day for three days. As a further refinement, it was deemed advisable to increase the sample portions for the main ditch during those periods when stump washer water was diverted through this plant outlet. Indications are that this stump washer water may increase the flow as much as 20 per cent.

7. Hydraulics Measurement

The recorded flow of 800 g.p.m. for the stump washer water was to be checked. The discharge through the main ditch was at no time increased by as much as 800 g.p.m., indicating that the stump washer water may be somewhat less than this volume. A further discrepancy appears in the solids data for stump washer water compared with the main ditch effluent for February 14 and February 16, which days the stump washer water was diverted through the main ditch. A lower water consumption for the stump washer will serve to bring these solids data more nearly in line.

8. Tank Car Washings

The dresinate tank car which was washed on February 14 gave a waste having a population equivalent of approximately 6,000. Considering the short period in which this waste is discharged through the plant sewers to Bowie River, this operation represents a shock load on the receiving stream. This incident serves to emphasize the importance of tank car washings as related to the pollutional effect of wastes from this plant. It is understood that tank cars are drained to receptacles initially prior to initiating cleaning. The importance of this practice should be impressed on the workmen as a means of minimizing this waste source. It would seem that this recovered material should have some value.

9. Phenol Determination

Company representatives expressed an interest in determining phenol on wastes from the synthetic plant and the main ditch effluent. Studies made by Hercules Powder Company on wastes from one of their plants located in the Philadelphia area indicated the presence of phenolic type compounds. It was pointed out that substances would be expected in these wastes which might interfere with the phenol determination. This condition will add to the amount of work required for phenol analysis. It was concluded that this study would not be complete without examination for phenol on wastes from the synthetic plant and the main ditch effluent. Separate samples will be collected and preserved for the phenol determination for three different days.

10. Process Water

The plant river water intake is located downstream from the stump washer discharge. There is a good possibility that the stump washer water will impart some 5-day B.O.D. and solids to the process water. It was decided to take river water samples from the pump discharge for analysis in order that appropriate deductions may be made from the plant wastes.

11. Stream Studies

Hercules Powder Company officials are interested in correlating wastes from their Hattiesburg plant with existing conditions in Bowie River. Since the time of flow in Bowie River between Hercules plant discharge and confluence of Leaf River is only a few hours, it is evident that these wastes will likewise contribute pollution to Leaf River. It was decided to establish sampling points on Bowie River at significant points above and below the Hercules plant, as well as in Leaf River

above and below confluence of Bowie River. This sampling program will provide data which will serve as basis for deciding regarding a more comprehensive stream survey of Leaf River in the Hattiesburg, Mississippi, area.

12. Sample Deletion

Data for two sampling points indicated relatively insignificant wastes. These were the acid concentrator and the drop tanks. The acid concentrator barometric condenser water had a relatively low 5-day B.O.D., the highest reported being 25 p.p.m. The drop tanks contributed only 104 gallons per day. The drop tank solution had a high 5-day B.O.D., the maximum being 5800 p.p.m. The pounds of 5-day B.O.D. in each case were relatively small. It was decided to do no further analytical work on samples from these two points.

13. Continuing Study

It was planned to complete the comprehensive sampling program inside Hercules naval stores plant during the week of February 26. It may prove desirable to perform further sampling for special studies. This will be determined on examination of the remaining data.

Sampling of Bowie River and Leaf River above and below confluence of Bowie River will be initiated during the week of March 4. The two Hercules naval stores outfall sewers will be sampled in conjunction with the stream studies. The stream study may include bottom sampling if it appears that such samples will contribute useful information.

14. Summary

Based on examination of analytical data for the period February 14 to February 23, inclusive, it appears that ten days results for Hercules Naval Stores, Hattiesburg plant, will give a reasonably accurate picture of the pollutional effect of the wastes from this industry and will indicate the principal sources of these pollutional materials. Work planned on the receiving waters will provide additional data essential to evaluation of this industrial waste - stream pollution problem.

Hayse H. Black
Chief, Industrial Wastes Section
Survey and Investigation Branch.

HHB/ecf

DICHROMATE REFLUX METHOD FOR DETERMINATION OF OXYGEN CONSUMED

Special Apparatus

1. Reflux apparatus, consisting of 300-ml., round-bottomed flask with a 24/40 taper-joint neck connected with a Friedrich's reflux condenser.

Reagents and Solutions

1. Potassium dichromate, 0.2500 N
2. Sulfuric acid, concentrated
3. Ferrous ammonium sulfate, 0.2500 N
4. Indicator, o-phenanthroline ferrous complex

Procedure

1. Dilute an appropriate amount of sample to 50 ml. with distilled water in a 300-ml. round-bottomed flask with a taper joint neck.
2. Add 25 ml. of 0.2500 N potassium dichromate and 75 ml. of concentrated sulfuric acid. For the best quantitative results, use 50 per cent by volume sulfiric acid.
3. Reflux for two hours.
4. Cool, transfer the mixture to an Erlenmeyer flask and titrate the excess potassium dichromate with approximately 0.2500 N ferrous ammonium sulfate, using o-phenanthroline ferrous complex as an indicator.
5. Reflux a blank at the same time, using the same amount of reagents and substituting 50 ml. of distilled water for the sample.
6. The ferrous ammonium sulfate must be standardized daily.

Calculations

$$\text{O.C., p.p.m.} = \frac{(a-b) \times \text{normality (standard Fe)} \times 8000}{\text{Volume of Sample}}$$

where, O.C. = Oxygen Consumed

a = ml. of ferrous ammonium sulfate used for blank

b = ml. of ferrous ammonium sulfate used for sample.

DETERMINATION OF OIL (ETHER-EXTRACTABLE MATERIAL)

Sampling Procedure

1. Sampling Vessels

   (1) The sampling vessel shall be a clean glass-stoppered vessel of known capacity of approximately 1 liter.

   (1-a) If it is desired to examine composite samples, vessels shall consist of clean glass-stoppered vessels of known capacity. The total volume held by the number of portions composited shall be approximately 1 liter.

2. Extraction mixture, 50% C.P., fat-free, carbon tetrachloride by volume, 50% C.P. A.C.S. diethyl ether by volume.

3. Sample portions shall consist of a full bottle, or a vessel filled up to a prefixed calibration mark. The entire contents of a sample vessel must be used.

Procedure

Determine the pH of the sample. If compositing is being practiced, the subsamples should be composited in a 2-liter separatory funnel and mixed before the pH is determined. If the pH is above 6.0, add 1 ml. portions of concentrated HCl acid until the pH falls below 6.0. Then, add a further 10 ml. portion of concentrated HCl acid. If the pH of the sample is initially below 6.0, add 10 ml. of concentrated HCl acid without preliminary adjustment of pH.

Rinse the sample container, or containers, with an 80 ml. portion of the extraction mixture. After the extraction mixture has been used to rinse the sample containers, transfer it to the extraction flask and shake vigorously for two minutes. The sample is allowed to stand long enough for the solvent layer to separate. The lower solvent layer is then separated. Rinse the sample container, or containers, with a further 60 ml. portion of solvent. Add the solvent to the sample in the separatory funnel and shake for two minutes. Separate the solvent layer and add it to the solvent already collected from the first extraction. Make a third rinsing and extraction, using a further 60 ml. portion and the same technique as was used for the second extraction.

Put the combined extract portions into the separatory funnel with 1 liter of distilled water and shake thoroughly for two minutes. Separate the extract, and wash the distilled water with 30 ml. of extraction mixture. Add the wash solvent to the separated extract. Filter the combined extract portions through a fat-free filter paper. Wash the paper three times with 5 ml. portions of solvent combining the filtrate and the wash solvent. Evaporate the combined extract on a steam bath under a hood in a weighed dish. When the solvent has been evaporated off, transfer the dish to an oven maintained at 103°C. Dry for 30 minutes, cool the dish in a desiccator. Reweigh the dish.

Calculations

$$\text{Ether-Extractable Material, p.p.m.} = \frac{\text{Weight of extract in mg.}}{\text{Volume of sample in liters}}$$

DETERMINATION OF PHENOL

1. Sample Preservation and Handling Prior to Analysis

At the time the sample is collected, a 10% solution of $CuSO_4 \cdot 5H_2O$ shall be added at the rate of 10 ml. per liter of sample. Analysis of the sample as described below should be started the day the sample is collected, and in no case shall a sample be held in excess of 24 hours before analysis is begun.

2. Scope of Method

The procedure given is specifically designed to remove a multitude of interfering substances which may be present in industrial wastes, and for this reason is more complicated than would be required for normal aerobic stream samples.

Materials

(1) Distilled water - phenol and chlorine free.

(2) Borate buffer

3.1 g. boric acid (H_3BO_3)
3.5 g. KCl
32 ml. of 1 N NaOH

Make up to 1 liter with distilled water. Make up 5 ml. of the above buffer solution to 100 ml. with freshly distilled water and if the pH is not 9.4, add further caustic until 5 ml. of the buffer solution produces a pH of 9.4 when made up to 100 ml. with distilled water.

(3) Gibbs reagent: (Alcoholic concentrate)
Dissolve 0.2 g. of 2,6-dibromquinonechlorimide in 50 ml. of ethyl alcohol. Filter out any residue. If refrigerated, this reagent may be used for periods up to one week.

(4) Gibbe reagent: (Aqueous dilute solution).
Dilute 4.5 ml. of the alcoholic concentrate to 100 ml., use within 30 minutes, and preferably immediately.

Special Apparatus

(1) Spectrophotometer or electrophotometer capable of accommodating a cell of at least 5 cm. length which can be satisfactorily filled with 60 c.c. of liquid or less. Further, wave length selection of the light used shall be such that the instrument can operate with a wave length of 670 millimicrons and a slit width or wave length spread of not over 15 millimicrons, preferably of 2 millimicrons or less.

(2) Distillation Apparatus
1 liter Pyrex No. 3360 distilling apparatus with Graham condenser, or equivalent.

Details of Procedure

(1) To 606 c.c. of the sample treated with copper sulfate solution, add 1.5 ml. of 10% H_3PO_4. Check the pH of the sample, and if it is not below 4.0, add sufficient 10% H_3PO_4 to bring the pH below 4.0.

Using an all-glass still and condenser, such as a 1-liter Pyrex No. 3360 distilling flask with Graham condenser, the sample is distilled. 550 c.c. are distilled over rapidly. The distillation is then halted and 50 c.c. of chlorine and phenol-free distilled water are added to the residue in the distilling flask. A further 50 c.c. are distilled over more slowly in order to avoid cracking the distilling flask. The total distillate collected is 600 c.c. Acidify

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the distillate with 1 c.c. of concentrated H_3PO_4 and add 5 c.c. of 10% $CuSO_4$. Place the sample in 1-liter separatory funnel. Add 125 c.c. of C.P., ACS diethyl ether and shake the sample with the ether thoroughly. Separate the sample from the ether layer and make a second extraction of the sample with 125 ml. of ether, again separating the ether layer and combining it with the ether separated after the first extraction. Discard the water phase. Extract the ether sequentially with two 75 c.c. portions of N/10 NaOH. Combine both alkaline extracts of the ether, discarding the ether after the second extraction. Cautiously heat the alkaline extract under a hood, using an electric heater. Bring to a boil, and boil off sufficient liquor to reduce the volume to approximately 100 c.c. Add 2 c.c. of concentrated phosphoric acid, 5 c.c. of 10% $CuSO_4$ and make up the volume to 300 c.c. Using the type of distillation apparatus previously described, distill over 250 c.c. at a rapid rate. Halt the distillation. Add 50 c.c. of distilled water. Distill over a further 50 c.c. at a slower rate to bring the total volume collected up to 300 c.c.

Control portions should be prepared as follows:

(a) Blank - 300 c.c. of distilled water.

(b) Phenol control - 5 c.c. of a 1000 p.p.m. solution of phenol in water are diluted up to 1 liter. 10 c.c. of this dilution are made up to 1 liter to yield a 50 p.p.b. solution (0.05 p.p.m.). To 600 c.c. of this solution, 6 c.c. of $CuSO_4$ are added, and this control portion is treated exactly as is a sample under examination.

Place all samples, the phenol control, and the blank control, in glass-stoppered vessels of similar shape. Adjust the temperature of all portions so that all portions show less than 1°C. difference from the temperature of the phenol control, and the temperature is under 28°C. Add 15 ml. of borate buffer to each portion under examination and mix well by shaking. Add to each portion 5 ml. of freshly prepared dilute Gibbs reagent (aqueous dilute solution) and shake well immediately. Allow the samples to stand from 6 to 24 hours under identical temperature conditions, below 28°C.

To each portion add 75 ml. of n-butyl alcohol and shake thoroughly. Separate the water and n-butyl alcohol layers, using 500 ml. separatory funnels. Filter the alcoholic dye extract through a 18.5 cm. fluted paper.* After the paper is drained completely, wash once with

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* Filter paper should be washed with distilled water, made slightly alkaline with ammonium hydroxide, before filtration of the n-butyl extract.

a measured 5 ml. portion of n-butyl alcohol, combining the two portions of the filtrate. Make all of the alcoholic extract up to exactly 60 ml. with n-butyl alcohol and mix each portion by gentle swirling.

The optical density of all portions, including the control phenol sample, is now determined using a spectrophotometer or an electrophotometer, the sample being examined at a wave length of 670 millimicrons, with cells of not less than 5 cm. in length of light path, and a slit opening of not more than 15 millimicrons, and preferably not more than 1 millimicron. The samples and phenol standard are compared against the control blank as a reference.

Calculation:

$$\text{Phenol, p.p.b.} = \frac{50 . \text{Optical Density of Sample (670 millimicrons)}}{\text{Optical Density of Control Phenol (670 millimicrons)}}$$

If the optical density of the sample is greater than that of the control phenol sample, phenol shall be reported as "over 50 p.p.b."

Hattiesburg Naval Stores Survey
Hattiesburg, Mississippi
Data Summary Sheet
United States Public Health Service

SAMPLE POINT: Stump Washer Effluent

Lab. No.	Date 1951	Average Discharge GPM	MGD #	B.O.D. at 20°C. 5 Day ppm	B.O.D. at 20°C. 5 Day Lbs/day	Oxygen Consumed ppm	pH	Total Acidity ppm	Color ppm	Total Solids: Total ppm	Total Solids: Total Lbs/day	Total Solids: Volatile ppm	Total Solids: Volatile Lbs/day	Total Solids: Fixed ppm	Total Solids: Fixed Lbs/day	Suspended Solids: Total ppm	Suspended Solids: Total Lbs/day	Suspended Solids: Volatile ppm	Suspended Solids: Volatile Lbs/day	Suspended Solids: Fixed ppm	Suspended Solids: Fixed Lbs/day
11	2/18	450	0.432	-	-	8730	4.5	48	550	9160	33000	4490	16000	4780	17000	7040	25300	3010	10800	4030	14500
19	2/16	450	0.432	210	756	5720	4.7	58	900	12000	43000	5800	19500	6600	23500	9940	35800	4830	15300	5710	20600
28	2/19	450	0.432	152	547	4010	4.9	70	700	8700	31300	4400	15100	4300	16200	6880	24800	3000	10800	3880	14000
[illegible]	2/21	450	0.432	221	795	6320	4.7	64	900	20900	75200	14400	50400	6900	24800	15500	55800	13000	46800	2500	9000
50	2/22	450	0.432	162	583	3280	5.1	48	600	7800	28100	2240	7550	5700	20550	7340	26400	2090	7520	5250	18880
61	2/23	450	0.432	228	820	6080	4.9	28	900	10000	36000	3300	11900	6700	24100	9780	35200	3310	11900	6470	23300
70	2/26	450	0.432	184	662	7040	5.2	53	500	12280	43900	9300	33500	2900	10400	5380	19400	2530	9100	2850	10300
80	2/28	450	0.432	166	597	6730	5.0	34	1100	9500	34200	3300	11900	6200	22300	9050	32600	3070	11100	5980	21500
91	3/1	450	0.432	100	360	4160	4.9	46	700	7100	25600	2300	8280	4800	17320	7050	25300	2270	8170	4760	17130
103	3/2	450	0.432	248	892	5250	4.9	41	-	13800	49700	4400	16200	9300	33500	13580	48600	4090	15400	9210	33200
Mean		450	0.432	186	668	5730		51		11100	40000	5140	19000	5810	21000	9140	32900	4080	14700	5060	18200
Maximum				248	892	7040	5.2	70	1100	20900	75200	14400	50400	9300	33500	15500	55800	13000	46800	9210	33200
Minimum				100	360	3280	4.5	28	500	7100	25600	2100	7550	2900	10400	5380	19400	2090	7520	2580	9000

Stump Washer operates 16 hours a day.

All samples were 6 hour composites (10 AM - 3 PM) except Nos. 28 and 70 which were 8 hour composites (8 AM - 3 PM).

SAMPLE POINT Main Plant Effluent

Hercules Naval Stores Survey
Hattiesburg, Mississippi
Data Summary Sheet
United States Public Health Service

Lab. No.	Date 1951	Average Discharge GPM	MGD	B.O.D. at 20°C. 5 Day ppm	B.O.D. at 20°C. 5 Day Lbs/Day	Oxygen Consumed ppm	pH	Total Alkalinity ppm	Color ppm	Ether Extractable Material ppm	Ether Extractable Material lbs/day	Total Solids Total ppm	Total Solids Total lbs/day	Total Solids Volatile ppm	Total Solids Volatile lbs/day	Total Solids Fixed ppm	Total Solids Fixed lbs/day	Suspended Solids Total ppm	Suspended Solids Total lbs/day	Suspended Solids Volatile ppm	Suspended Solids Volatile lbs/day	Suspended Solids Fixed ppm	Suspended Solids Fixed lbs/day
37	2/19	3230	4.65	124	4800	345	6.9	42	100	39.8	1540	350	13560	113	4380	237	9180	96	3720	48	1860	48	1860
49	2/21	3310	4.77	290	11500	587	7.8	63	225	66.0	2620	414	16450	206	8190	208	8260	98	3890	74	2940	24	950
59	2/22	3470	5.00	193	8040	525	8.3	130	165	75.3	3140	470	19580	178	7080	300	12500	218	9080	108	4500	110	4580
69	2/23	3470	5.00	193	8040	530	8.7	92	190	99.2	4130	406	16910	180	7500	226	9410	-	-	-	-	-	-
79	2/26	3495	5.03	270	11300	665	7.1	93	200	119.0	4990	404	16930	170	7120	234	9810	162	6790	98	4110	64	2680
88	2/28	3660	5.27	261	11500	517	10.8	220	255	49.7	2180	558	24500	230	10100	328	14400	126	5530	62	2720	64	2810
97	3/1	3635	5.23	165	7190	372	8.3	79	115	32.8	1430	364	15840	132	5750	232	10110	132	5750	46	2000	86	3750
109	3/2	3465	4.99	370	15400	623	7.3	80	-	23.3	970	326	13550	134	5570	192	7980	104	4320	58	2410	46	1910
116	3/5	3040	4.38	338	12300	688	8.6	74	-	74.3	2710	396	14450	160	5840	236	8610	112	4090	60	2190	52	1900
119	3/6	3370	4.85	254	10300	568	6.7	41	-	62.0	2500	385	15550	140	5660	245	9890	148	5980	71	2870	77	3110
123	3/7	3315	4.77	-	-	424	10.9	220	-	49.2	1950	613	24360	217	8620	396	15740	142	5640	59	2340	83	3300
	Mean	3405	4.90	246	10000	531		100		62.8	2560	426	17430	168	6890	258	10540	134	5480	68	2790	66	2690
	Maximum	3660	5.27	370	15400	688	10.9	220	255	119.0	4990	613	24500	230	10100	396	15740	218	9080	108	4500	110	4580
	Minimum	3040	4.38	124	4800	345	6.7	41	100	23.3	970	326	13550	113	4380	192	7980	96	3720	46	1860	24	950

All samples were 8 hour composites (8 AM - 3 PM)

SAMPLE POINT Drop Tanks

Hercules Naval Stores Survey
Hattiesburg, Mississippi
Data Summary Sheet
United States Public Health Service

Lab. No.	Date 1951	Average Discharge MGD	B.O.D. at 20°C. 5-Day ppm	B.O.D. at 20°C. 5-Day Lbs/Day	Oxygen Consumed ppm	pH	Acidity T-Total M-Mineral ppm	Color ppm	Ether Extractable Material ppm	Ether Extractable Material Lbs/Day	Total Solids: Total ppm	Total Solids: Total lbs/day	Total Solids: Volatile ppm	Total Solids: Volatile lbs/day	Total Solids: Fixed ppm	Total Solids: Fixed lbs/day	Suspended Solids: Total ppm	Suspended Solids: Total lbs/day	Suspended Solids: Volatile ppm	Suspended Solids: Volatile lbs/day	Suspended Solids: Fixed ppm	Suspended Solids: Fixed lbs/day
9	2/14	0.000104	2040	1.8	8600	3.8	590T 130M	-	547	0.5	4180	3.6	3970	3.4	210	0.2	276	0.24	242	0.21	34	0.03
20	2/16	0.000104	4280	3.7	11100	4.1	930T 100M	5000	573	0.5	6860	5.9	6310	5.5	550	0.4	274	0.24	260	0.23	14	0.01
29	2/19	0.000104	5800	5.0	13800	4.0	920T 350M	3000	1120	1.0	6580	5.5	6080	5.2	380	0.3	1190	1.03	1180	1.02	10	0.01
41	2/21	0.000104	3300	2.9	9230	3.9	840T - M	3000	496	0.4	5070	4.4	4730	4.1	340	0.3	364	0.32	342	0.30	22	0.02
51	2/22	0.000104	4100	3.6	9560	4.2	870T 150M	2500	800	0.7	4960	4.	4610	4.0	350	0.3	694	0.60	658	0.57	36	0.03
62	2/23	0.000104	5080	4.4	9850	3.9	880T 290M	3000	417	0.4	5180	4.5	4840	4.2	340	0.3	614	0.53	596	0.52	18	0.01
71	2/26	0.000104	4160	3.6	9590	3.9	880T 160M	2000	-	-	4950	4.3	4670	4.0	280	0.3	750	0.65	722	0.63	28	0.02
Mean		0.000104	4110	3.6	10200		840T 200M		660	0.6	5370	4.6	5080	4.3	350	0.3	595	0.52	571	0.50	24	0.02
Maximum			5800	5.0	13800	4.2	930T 350M	5000	1120	1.0	6860	5.9	6310	5.5	550	0.4	1190	1.03	1180	1.02	36	0.03
Minimum			2040	1.8	8600	3.8	590T 100M	2000	417	0.4	4180	3.6	3970	3.4	210	0.2	274	0.24	242	0.21	10	0.01

All samples were collected in the morning as drop tanks were drained.

Hercules Naval Stores Survey
Hattiesburg, Mississippi
Data Summary Sheet
United States Public Health Service

SAMPLE POINT 7th Street Drainage Ditch *

Lab. No.	Date 1951	Time Sample Collected	Average Discharge# GPM	Average Discharge# MGD	B.O.D. at 20°C. 5 Day ppm	B.O.D. at 20°C. 5 Day Lbs/Day	Oxygen Consumed ppm	pH	Total Solids: Total ppm	Total Solids: Total lbs/day	Total Solids: Volatile ppm	Total Solids: Volatile lbs/day	Total Solids: Fixed ppm	Total Solids: Fixed lbs/day	Suspended Solids: Total ppm	Suspended Solids: Total lbs/day	Suspended Solids: Volatile ppm	Suspended Solids: Volatile lbs/day	Suspended Solids: Fixed ppm	Suspended Solids: Fixed lbs/day
228	3/30	10:00 AM	175	0.252	234	491	-	3.7	-	-	-	-	-	-	-	-	-	-	-	-
230	4/2	11:00 AM	175	0.252	130	273	143	6.7	130	273	49	103	81	170	28	59	8	17	20	42
239	4/3	9:30 AM	175	0.252	169	355	454	9.1	218	458	93	195	125	263	54	113	42	88	12	25
247	4/4	10:00 AM	175	0.252	172	361	226	7.0	147	309	64	134	83	175	35	73	25	52	10	21
255	4/5	9:30 AM	175	0.252	117	246	-	9.3	-	-	-	-	-	-	-	-	-	-	-	-
Mean			175	0.252	164	345	274		165	347	69	144	96	203	39	82	25	52	14	30
Maximum					234	491	454	9.3	218	458	93	195	125	263	54	113	42	88	20	42
Minimum					117	246	143	3.7	130	273	49	103	81	170	28	59	8	17	10	21

* Surface run-off, also includes pilot plant and laboratory waste water.

\# Dry weather flow.

All results based on grab samples collected at indicated time.

Hercules Naval Stores Survey
Hattiesburg, Mississippi
Data Summary Sheet
United States Public Health Service

SAMPLE POINT 731 Plant (Miscellaneous) *

Lab. No.	Date 1951	Time Sample Collected	Average Discharge GPM	MGD	BOD at 20°C. 5 Day ppm	BOD at 20°C. 5 Day lbs/day	Oxygen Consumed ppm	pH	Alkalinity T-Total P-Phenolphthalein ppm	Total Solids: Total ppm	Total Solids: Total lbs/day	Total Solids: Volatile ppm	Total Solids: Volatile lbs/day	Total Solids: Fixed ppm	Total Solids: Fixed lbs/day	Suspended Solids: Total ppm	Suspended Solids: Total lbs/day	Suspended Solids: Volatile ppm	Suspended Solids: Volatile lbs/day	Suspended Solids: Fixed ppm	Suspended Solids: Fixed lbs/day
235	4/2	11:00 AM	8.50	0.012	)3860	-	-	11.9	-	12500	1275	9100	928	3400	347	372	38	340	35	32	3
243	4/3	10:00 AM	8.50	0.012	)15800	-	-	9.5	-	16600	1690	13800	1410	2800	280	6330	645	6310	643	20	2
249	4/4	10:00 AM	8.50	0.012	)15400	-	-	8.5	-	-	-	-	-	-	-	-	-	-	-	-	-
407	4/18	9:00 AM	8.50	0.012	20200	2060	45200	9.8	3200 T 150 P	-	-	-	-	-	-	-	-	-	-	-	-
412	4/19	9:00 AM	8.50	0.012	45000	4590	94800	9.8	4260 T 650 P	-	-	-	-	-	-	-	-	-	-	-	-
415	4/20	9:00 AM	8.50	0.012	49000	5000	96900	9.6	3100 T 350 P	-	-	-	-	-	-	-	-	-	-	-	-
Mean			8.50	0.012	38100	3880	79000		3520 T 380 P	14600	1480	11400	1170	3100	310	3350	341	3325	339	25	2
Maximum					49000	5000	96900	11.9	4260 T 650 P												
Minimum					20800	2060	45200	8.5	3100 T 150 P												

* Includes floor wash, accidental spills, oil tank vents, etc.
) Indicates a value greater than – not included in averages.
All results based on grab samples collected at indicated time.

Hercules Naval Stores Survey
Hattiesburg, Mississippi
Data Summary Sheet
United States Public Health Service

SAMPLE POINT 731 (Dehydrogenation) Plant

Lab. No.	Date 1951	Average Discharge GPM	MGD	BOD at 20°C 5 Day ppm	BOD at 20°C 5 Day lbs/day	Oxygen Consumed ppm	pH	Total Alkalinity ppm	Ether Extractable Material ppm	Ether Extractable Material lbs/day	Total Solids Total ppm	Total Solids Total lbs/day	Total Solids Volatile ppm	Total Solids Volatile lbs/day	Total Solids Fixed ppm	Total Solids Fixed lbs/day	Suspended Solids Total ppm	Suspended Solids Total lbs/day	Suspended Solids Volatile ppm	Suspended Solids Volatile lbs/day	Suspended Solids Fixed ppm	Suspended Solids Fixed lbs/day
14	2/14	210	0.302	120	302	459	6.0	11	90	226	265	667	112	282	153	385	39	98	28	70	11	28
23	2/16	220	0.317	290	766	604	6.1	11	51	135	157	415	76	201	81	214	58	153	36	95	22	58
32	2/19	220	0.317	86	227	181	6.5	13	40	106	92	243	42	111	50	132	25	66	11	30	14	36
45	2/21	220	0.317	336	887	645	6.5	21	59	156	141	372	97	256	44	116	49	130	31	82	18	49
54	2/22	220	0.317	304	803	694	6.2	17	90	238	134	354	59	156	75	198	50	132	37	98	13	34
65	2/23	220	0.317	423	1120	673	-	-	93	246	195	515	121	320	74	195	49	129	18	48	31	82
74	2/26	220	0.317	365	964	745	6.3	13	113	298	130	343	75	198	55	145	51	135	44	116	7	19
83	2/28	285	0.410	179	611	606	6.0	16	99	338	132	450	98	266	54	184	35	120	21	72	14	48
94	3/1	240	0.346	282	813	632	6.2	14	76	219	124	357	63	182	61	175	14	40	10	29	4	11
106	3/2	250	0.360	179	536	684	5.9	12	79	237	130	390	67	201	63	189	40	120	31	93	9	27
Mean		230	0.332	256	703	592		14	79	220	150	411	79	217	71	194	41	112	27	73	14	39
Maximum		285	0.410	423	1120	684	6.5	21	113	338	265	667	121	320	153	385	58	153	44	116	31	82
Minimum		210	0.302	86	227	181	5.9	11	40	106	92	243	42	111	44	116	14	40	10	29	4	11

All samples were 8 hour composites (8 AM - 3 PM).

SAMPLE POINT Solvent Recovery

Hercules Naval Stores Survey
Hattiesburg, Mississippi
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United States Public Health Service

Lab. No.	Date 1951	Average Discharge GPM	MGD	BOD at 20°C. 5 Day ppm	lbs/day	Oxygen Consumed ppm	pH	Acidity T-Total M-Mineral ppm	Ether Extractable Material ppm	lbs/day	Total Solids: Total ppm	lbs/day	Volatile ppm	lbs/day	Fixed ppm	lbs/day	Suspended Solids: Total ppm	lbs/day	Volatile ppm	lbs/day	Fixed ppm	lbs/day
12	2/14	80	0.115	1780	1710	2560	3.4	290T 120M	34	32	135	129	104	100	31	29	20	19	15	14	5	5
21	2/16	80	0.115	1950	1870	2980	3.3	290T 170M	34	32	157	150	110	105	47	45	28	27	20	19	8	8
30	2/19	80	0.115	1840	1760	2370	3.4	330T 160M	28	27	178	171	145	139	33	32	13	12	12	11	1	1
43	2/21	80	0.115	1840	1760	2440	3.3	320T 180M	18	17	136	130	112	107	24	23	17	16	14	13	3	3
52	2/22	80	0.115	2400	2300	3280	3.4	300T 180M	34	32	173	166	124	119	49	47	17	16	13	12	4	4
63	2/23	80	0.115	2580	2470	2500	3.3	340T 200M	28	27	148	142	114	109	34	33	16	15	12	11	4	4
72	2/26	80	0.115	2730	2620	3010	3.3	340T 190M	27	26	146	140	103	99	43	41	16	15	12	11	4	4
81	2/28	80	0.115	2350	2250	3400	3.3	310T 190M	27	26	149	143	104	100	45	43	12	11	8	7	4	4
92	3/1	80	0.115	1970	1890	2690	3.4	270T 180M	44	42	113	108	75	72	38	36	11	10	5	5	6	5
104	3/2	80	0.115	1410	1350	2700	3.5	250T 140M	30	29	152	146	120	115	32	31	14	13	12	11	2	2
Mean		80	0.115	2080	2000	2790		300T 170M	30	29	150	143	111	107	39	36	16	15	12	11	4	4
Maximum				2730	2620	3400	3.5	340T 200M	44	42	178	171	145	139	49	47	28	27	20	19	8	8
Minimum				1410	1350	2370	3.3	250T 120M	18	17	113	108	75	72	24	23	11	10	5	5	1	1

All samples were 8 hour composites (8 AM - 3 PM).

SAMPLE POINT: Synthetics Plant

Hercules Naval Stores Survey
Hattiesburg, Mississippi
Data Summary Sheet
United States Public Health Service

Lab. No.	Date 1952	Average Discharge GPM	MGD	BOD at 20°C 5 Day ppm	BOD at 20°C 5 Day lbs/day	Oxygen Consumed ppm	pH	Alkalinity P-Total P-Phenolphthalein ppm	Color ppm	Ether Extractable Material ppm	Ether Extractable Material lbs/day	Total Solids Total ppm	Total Solids Total lbs/day	Total Solids Volatile ppm	Total Solids Volatile lbs/day	Total Solids Fixed ppm	Total Solids Fixed lbs/day	Suspended Solids Total ppm	Suspended Solids Total lbs/day	Suspended Solids Volatile ppm	Suspended Solids Volatile lbs/day	Suspended Solids Fixed ppm	Suspended Solids Fixed lbs/day
17	2/14	365	0.526	135	592	321	10.1	310T 14P	100	45	197	123	539	69	302	54	237	31	136	23	101	8	35
26	2/16	400	0.576	179	859	767	10.5	1230T 105P	80	166	796	380	1820	247	1180	133	640	190	912	145	696	45	216
36	2/19	405	0.583	132	641	405	9.4	87T 0P	175	54	262	213	1050	115	568	98	482	56	275	86	426	10	49
48	2/21	405	0.583	250	1210	702	9.6	225T 110P	150	238	1160	484	2350	287	1390	217	1050	69	306	52	253	11	53
58	2/22	445	0.641	334	1780	1130	10.5	950T 530P	-	345	1840	918	4900	358	2880	566	3020	384	2050	346	1850	38	200
68	2/23	440	0.634	383	2020	958	10.8	585T 405P	-	334	1760	563	2970	408	2120	161	848	32	169	28	148	4	21
78	2/26	450	0.648	692	3740	1700	9.9	1230T 910P	-	820	4430	381	2060	305	1650	76	410	243	1310	234	1260	9	50
87	2/28	425	0.598	209	1020	888	10.1	385T 255P	-	656	3270	316	1570	227	1080	99	490	103	513	91	453	12	60
96	3/1	425	0.612	131	668	987	9.7	108T 0P	-	370	1890	538	2740	434	2210	104	530	182	928	148	856	34	72
108	3/2	414	0.598	269	1340	445	10.2	475T 360P	-	91	453	238	1190	113	560	125	630	34	169	23	115	11	54
Mean		416	0.600	271	1390	830		560T 370P		312	1600	415	2120	252	1280	163	840	130	667	114	586	16	81
Maximum		445	0.641	692	3740	1700	10.5	1230T 1050P	175	820	4430	918	4900	498	2210	566	3020	384	2050	346	1850	45	216
Minimum		365	0.526	131	641	321	9.4	87T 0P	80	45	197	123	539	69	302	54	237	31	136	23	101	4	21

All samples were 8 hour composites (8 AM - 5 PM)

SAMPLE POINT Furfuraldehyde Recovery

Hercules Naval Stores Survey
Hattiesburg, Mississippi
Data Summary Sheet
United States Public Health Service

Lab. No.	Date 1951	Average Discharge GPM	MGD	BOD at 20°C. 5 Day ppm	lbs/day	Oxygen Consumed ppm	pH	Acidity T-Total M-Mineral ppm	Color ppm	Ether Extractable Material ppm	lbs/day	Total Solids: Total ppm	lbs/day	Volatile ppm	lbs/day	Fixed ppm	lbs/day	Suspended Solids: Total ppm	lbs/day	Volatile ppm	lbs/day	Fixed ppm	lbs/day
13	2/14	6.67	0.0096	8600	688	14900	3.4	230T 172M	100	65	5.2	158	13	131	11	27	2	2.4	0.2	2.0	0.2	0.4	0.0
22	2/16	6.67	0.0096	11600	928	15800	3.4	220T 180M	80	14	1.1	192	15	161	13	31	2	14.4	1.2	9.2	0.8	5.2	0.4
31	2/19	6.67	0.0096	10400	832	13600	3.5	180T 120M	60	10	0.8	114	9	89	7	25	2	4.8	0.4	4.8	0.4	0.0	0.0
44	2/21	6.67	0.0096	11900	952	14900	3.4	170T 140M	100	4	0.3	138	11	112	9	26	2	8.4	0.7	8.4	0.7	0.0	0.0
53	2/22	6.67	0.0096	10800	864	14800	3.5	170T 100M	90	25	2.0	125	10	90	7	35	3	-	-	-	-	-	-
64	8/23	6.67	0.0096	14000	1120	15600	3.4	170T 110M	90	9	0.7	129	10	84	7	45	3	-	-	-	-	-	-
73	2/26	6.67	0.0096	12400	992	15200	3.5	240T 160M	100	-	-	154	12	115	9	39	3	-	-	-	-	-	-
82	2/28	6.67	0.0096	10500	840	15000	3.4	210T 150M	100	-	-	168	13	113	9	55	4	-	-	-	-	-	-
93	3/1	6.67	0.0096	10600	848	14500	3.5	190T 80M	100	-	-	141	11	94	8	47	3	-	-	-	-	-	-
105	3/2	6.67	0.0096	6150	492	11000	3.5	140T 120M	-	-	-	193	15	159	13	34	2	-	-	-	-	-	-
Mean		6.67	0.0096	10700	856	14500		190T 120M		21	1.7	151	12	115	9	36	3	7.5	0.6	6.1	0.5	1.4	0.1
Maximum				14000	1120	15800	3.5	240T 180M	100	65	5.2	192	15	161	13	55	4	14.4	1.2	9.2	0.8	5.2	0.4
Minimum				6150	492	11000	3.4	140T 8M	60	4	0.3	114	9	84	7	25	2	2.4	0.2	2.0	0.2	0.4	0.0

All samples were 8 hour composites (8 AM - 5 PM).

SAMPLE POINT: Poly-Pale Solvent Recovery

Hercules Naval Stores Survey
Hattiesburg, Mississippi
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United States Public Health Service

Lab. No.	Date 1951	Average Discharge GPM	Average Discharge MGD	B.O.D. at 20°C. 5 Day ppm	B.O.D. at 20°C. 5 Day lbs/day	Oxygen Consumed ppm	pH	Acidity T-Total M-Mineral ppm	Color ppm	Ether Extractable Material ppm	Ether Extractable Material lbs/day	Total Solids: Total ppm	Total Solids: Total lbs/day	Total Solids: Volatile ppm	Total Solids: Volatile lbs/day	Total Solids: Fixed ppm	Total Solids: Fixed lbs/day	Suspended Solids: Total ppm	Suspended Solids: Total lbs/day	Suspended Solids: Volatile ppm	Suspended Solids: Volatile lbs/day	Suspended Solids: Fixed ppm	Suspended Solids: Fixed lbs/day
15	2/14	225	0.324	26	70	380	2.0	840T 720M		27.8	75	2760	7450	2140	5780	620	1670	36	97	20	54	16	43
24	2/16	235	0.338	37	104	300	2.1	710T 600M		13.9	90	1330	3740	840	2360	490	1380	45	126	14	39	31	87
33	2/19	245	0.352	60	176	297	1.8	1200T 1130M		11.4	33	1440	4220	1330	3900	110	320	26	76	8	23	18	53
46	2/21	75	0.108	41	37	673	2.0	720T 690M		11.5	10	1380	1240	880	790	500	450	32	29	14	13	18	16
55	2/22	90	0.130	106	115	482	1.9	1130T 960M		17.0	18	1330	1440	495	540	835	900	31	34	14	15	17	19
66	2/23	100	0.144	95	114	773	2.3	750T 510M		10.1	12	1340	1610	575	690	765	920	24	28	9	10	15	18
75	2/26	90	0.130	158	171	606	1.7	2330T 1980M		12.8	14	2990	3240	2240	2430	750	810	19	20	13	14	6	6
84	2/28	205	0.295	35	86	223	2.5	380T 300M		5.3	13	1020	2510	363	890	657	1620	19	47	8	20	11	27
95	3/1	205	0.295	60	147	227	3.2	220T 140M		5.2	13	885	2170	203	500	682	1670	19	47	12	30	7	17
107	3/2	220	0.317	139	367	335	2.2	500T 460M		92.8	245	863	2280	529	1400	334	880	25	66	18	48	7	18
Mean		169	0.243	76	139	430		880T 750M		20.8	52	1530	3990	960	1930	570	1060	28	57	13	27	15	30
Maximum		245	0.352	158	367	773	3.2	2330T 1980M		92.8	245	2990	7450	2240	5780	835	1670	45	126	20	54	31	87
Minimum		75	0.108	26	37	223	1.7	220T 140M		5.2	10	863	1240	203	500	110	320	19	20	8	10	4	6

All samples were 8 hour composites (8 AM - 3 PM)

Hercules Naval Stores Survey
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SAMPLE POINT: Isomerization Wash Water

Lab. No.	Date 1951	Time Sample Collected	Source	Average Discharge Gallons/day	BOD at 20°C 5 Day ppm	BOD at 20°C 5 Day lbs/day	Oxygen Consumed ppm	pH	Alkalinity T-Total P-Phenolphthalein ppm	Phenols ppm	Phenols lbs/day	Total Solids Total ppm	Total Solids Total lbs/day	Total Solids Volatile ppm	Total Solids Volatile lbs/day	Total Solids Fixed ppm	Total Solids Fixed lbs/day	Suspended Solids Total ppm	Suspended Solids Total lbs/day	Suspended Solids Volatile ppm	Suspended Solids Volatile lbs/day	Suspended Solids Fixed ppm	Suspended Solids Fixed lbs
184	3/21	6:00 PM	First Wash	400	26500	88	16400	*	149000 T 119000 P	3120	10.4	-	-	-	-	-	-	-	-	-	-	-	-
209	3/23	8:00 PM	First Wash	400	18900	63	17400	*	87100 T 58800 P	1750	5.8	-	-	-	-	-	-	-	-	-	-	-	-
232	3/31	10:00 PM	First Wash	400	22800	76	24700	*	-	2250	7.5	153000	509	37800	126	115000	383	36	0.12	8	0.03	28	0.09
241	4/2	12:00 M	First Wash	400	11990	40	9750	*	-	1000	3.3	110000	366	50000	166	60000	200	28	0.09	4	0.01	24	0.08
257	4/5	4:00 AM	First Wash	400	20400	68	23400	*	-	1740	5.8	120000	400	25000	83	95000	317	160	0.53	88	0.29	72	0.24
Mean				400	20100	67	18300		116000 T 90000 P	1990	6.6	128000	425	37600	125	90000	300	75	0.25	33	0.11	42	0.14
408	4/16	10:00 PM	Second Wash	400	10300	34	13000	12.3	6370 T 3480 P														
416	4/20	5:00 AM	Second Wash	400	3600	29	13800	12.2	6990 T 3420 P														
Mean				400	9450	32	13400		6680 T 3450 P														

* Too alkaline to determine by available methods.

All results are based on grab samples collected at indicated time.

Hercules Naval Stores Survey
Hattiesburg, Mississippi
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SAMPLE POINT Acid Concentrator

Lab. No.	Date 1951	Average Discharge GPM	Average Discharge MGD	BOD at 20°C. 5-day ppm	BOD at 20°C. 5-day lbs/day	Oxygen Consumed ppm	pH	Acidity T-Total M-Mineral ppm
16	2/14	60	0.086	8.4	6.0	23.9	2.7	110 T 100 M
25	2/16	60	0.086	11.1	8.0	17.6	3.0	56 T 45 M
34	2/19	60	0.086	20.7	14.8	32.6	2.6	145 T 140 M
47	2/21	60	0.086	19.1	13.7	45.6	3.3	56 T 43 M
56	2/22	60	0.086	24.6	17.6	39.7	2.4	260 T 230 M
67	2/23	60	0.086	14.1	10.1	16.6	3.4	61 T 25 M
76	2/26	60	0.086	21.6	15.5	30.0	2.8	110 T 86 M
Mean		60	0.086	17.1	12.2	29.4		114 T 96 M
Maximum		60	0.086	24.6	17.6	45.6	3.4	260 T 230 M
Minimum		60	0.086	8.4	6.0	16.6	2.6	56 T 25 M

All samples were 8-hour composites (8 AM - 3 PM)

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SAMPLE POINT Hydrogenation Plant

Lab. No.	Date 1951	Average Discharge Gals/day	BOD at 20° C. 5 Day ppm	BOD at 20° C. 5 Day lbs/day	Oxygen Consumed ppm	pH	Ether Extractable Material ppm	Ether Extractable Material lbs/day	Total Solids: Total ppm	Total Solids: Total lbs/day	Total Solids: Volatile ppm	Total Solids: Volatile lbs/day	Total Solids: Fixed ppm	Total Solids: Fixed lbs/day	Suspended Solids: Total ppm	Suspended Solids: Total lbs/day	Suspended Solids: Volatile ppm	Suspended Solids: Volatile lbs/day	Suspended Solids: Fixed ppm	Suspended Solids: Fixed lbs/day
226	3/30	36	270000	81	-	*	-	-	437000	130	437000	130	0	0	31300	10	31300	10	0	0
234	4/2	36	388000	116	1 110 000	*	417 000	125	384000	115	384000	115	0	0	37100	11	37100	11	0	0
242	4/3	36	142000	43	334 000	*	586 000	176	409000	123	409000	123	0	0	11000	3	11000	3	0	0
253	4/4	36	726000	218	1 430 000	*	398 000	119	-	-	-	-	-	-	-	-	-	-	-	-
MEAN		36	382000	115	958 000		467 000	140	410000	123	410000	123	0	0	26500	8	26500	8	0	0

* No reaction using Beckman electrometric pH meter model G.
All results based on grab samples collected at 10:00 AM.

SAMPLE POINT Still House Oil Trap Effluent

Lab. No.	Date 1951	Average Discharge GPM	Average Discharge MGD	BOD at 20°C. 5 Day ppm	BOD at 20°C. 5 Day lbs/day	Oxygen Consumed ppm	pH	Ether Extractable Material ppm	Ether Extractable Material lbs/day	Total Solids: Total ppm	Total Solids: Total lbs/day	Total Solids: Volatile ppm	Total Solids: Volatile lbs/day	Total Solids: Fixed ppm	Total Solids: Fixed lbs/day	Suspended Solids: Total ppm	Suspended Solids: Total lbs/day	Suspended Solids: Volatile ppm	Suspended Solids: Volatile lbs/day	Suspended Solids: Fixed ppm	Suspended Solids: Fixed lbs/day
237	4/2	6.80	0.0098	1740	142	1820	4.2	10.2	0.8	202	16.5	112	9.4	90	7.1	44	3.6	8	0.6	36	3.0
244	4/3	6.80	0.0098	1270	104	1390	5.2	16.9	1.4	16	1.3	14	1.1	2	0.2	-	-	-	-	-	-
251	4/4	6.80	0.0098	1090	89	920	4.1	8.5	0.7	21	1.7	11	0.9	10	0.8	-	-	-	-	-	-
MEAN		6.80	0.0098	1370	112	1380		10.9	1.0	79	6.5	44	3.6	33	2.7	44	3.6	8	0.6	36	3.0

All results based on grab samples collected at 10:00 AM.

Hercules Naval Stores Survey
Hattiesburg, Mississippi
Date Summary Sheet
United States Public Health Service

SAMPLE POINT Tank Car Wash

Lab. No.	Date 1951	Type of Tank Car	Average Discharge Gallons	200 at 20°C. 5 day ppm	200 at 20°C. 5 day lbs/car	Oxygen Consumed ppm	pH	Alkalinity % total P-Phenolphthalein ppm	Ether Extractable Material ppm	Ether Extractable Material lbs/car	Total ppm	Total lbs/car	Volatile ppm	Volatile lbs/car	Fixed ppm	Fixed lbs/car	Total ppm	Total lbs/car	Volatile ppm	Volatile lbs/car	Fixed ppm	Fixed lbs/car
8	2/13	Dresinate	500	26500	110	57000	8.9	2740T 570P	23800	100	27500	115	24000	100	3500	15	264	1.0	100	0.4	164	0.6
10	2/14	Dresinate	500	248000	1030	705000	9.2	37800T 0P	332000	1380	337000	1400	274000	1140	63000	260	3000	12.5	3000	12.5	0.0	0.0
40	2/21	Asphalt	500	3000	12	5230	10.9	20200T 8500P	5240	22	25200	105	2500	10	22700	95	54	0.2	54	0.2	0.0	0.0
57	2/22	Benzol	500	24	0.1	70	11.1	2740T 1230P	7	0.03	3060	13	135	1	2925	12	17	0.07	10	0.04	7	0.03
77	2/26	Rosin	4000	28	0.9	56	8.5	33T 0P	19	0.6	115	4	50	2	65	2	-	-	-	-	-	-
85	2/28	Dark Size	500	2400	10	7190	12.3	6800T 5150P	1770	7	11900	50	230	1	11670	49	-	-	-	-	-	-
86	2/28	Asphalt	500	69	0.3	300	11.3	10500T 3790P	29	0.1	8900	37	1680	7	7220	30	-	-	-	-	-	-
101	3/2	Asphalt	500	45	0.2	74	11.1	6900T 2970P	15	0.06	7500	31	250	1	7250	30	-	-	-	-	-	-
102	3/2	Turpentine	500	485	2	995	11.3	11000T 4930P	150	0.6	12400	52	900	4	11500	48	-	-	-	-	-	-
MAXIMUM			4000	248000	1030	705000	12.3	37800T 8500P	332000	1380	337000	1400	274000	1140	63000	260	3000	12.5	3000	12.5	164	0.6
MINIMUM			500	24	0.1	56	8.5	33T 0P	7	0.03	115	4	50	1	65	2	17	0.07	10	0.04	7	0.03

All samples were grab samples collected at the tank cars were being drained.

Hercules Naval Stores Survey
Results of Special Studies
Summary Sheet
United States Public Health Service

Lab. No.	Date 1951	Source	Average Discharge GPM	Average Discharge MGD	Terpin Hydrate * ppm	Terpin Hydrate * lbs/day	B.O.D. at 20° C. 5 Day Containing Terpin Hydrate ppm	B.O.D. at 20° C. 5 Day Containing Terpin Hydrate lbs/day	B.O.D. at 20° C. 5 Day Terpin Hydrate Removed ppm	B.O.D. at 20° C. 5 Day Terpin Hydrate Removed lbs/day	Oxygen Consumed Containing Terpin Hydrate ppm	Oxygen Consumed Containing Terpin Hydrate lbs/day	Oxygen Consumed Terpin Hydrate Removed ppm	Oxygen Consumed Terpin Hydrate Removed lbs/day	Per Cent Reduction B.O.D.	Per Cent Reduction O.C.
81	2/25	Solvent Recovery	80	0.115	900	865	2,350	2,250	1,150	1,100	3,400	3,260	1,550	1,480	51	54
92	3/1		80	0.115	600	575	1,965	1,880	572	548	2,695	2,580	760	730	71	72
104	3/2		80	-0.115	600	575	1,410	1,350	508	486	2,700	2,590	790	760	64	71
		MEAN	80	0.115	700	672	1,910	1,830	743	711	2,930	2,810	1,030	990	62	66
82	2/28	Furfural-dehyde	6.67	0.0096	6,000	480	10,500	840	3,325	265	15,050	1,200	5,175	414	69	66
93	3/1	Recovery	6.67	0.0096	5,000	400	10,700	856	3,675	294	14,490	1,160	5,340	427	66	63
105	3/2		6.67	0.0096	3,500	280	6,000	480	2,920	234	11,050	880	4,620	369	51	58
		MEAN	6.67	0.0096	4,830	385	9,070	725	3,300	264	13,500	1,080	5,050	400	62	62

* Analyses made by Hercules Laboratory.
All samples were 8 hour composites (8 AM - 5 PM).

Lab. No.	Date 1951	Source	Average Discharge GPM	Average Discharge MGD	Dissolved Oils* ppm	Dissolved Oils* lbs/day	B.O.D. at 20° C. 5 Day Containing Dissolved Oil ppm	B.O.D. at 20° C. 5 Day Containing Dissolved Oil lbs/day	B.O.D. at 20° C. 5 Day Dissolved Oil Removed ppm	B.O.D. at 20° C. 5 Day Dissolved Oil Removed lbs/day	Oxygen Consumed Containing Dissolved Oil ppm	Oxygen Consumed Containing Dissolved Oil lbs/day	Oxygen Consumed Dissolved Oil Removed ppm	Oxygen Consumed Dissolved Oil Removed lbs/day	Per Cent Reduction B.O.D.	Per Cent Reduction O. C.
237	4/2	Still House Oil Trap Effluent	6.80	0.0098	500	40	1,740	142	900	73	1,820	148	762	62	48	58
244	4/3		6.80	0.0098	300	24	1,270	104	260	22	1,390	113	222	18	80	84
251	4/4		6.80	0.0098	200	16	1,090	89	300	24	910	75	257	21	72	72
		MEAN	6.80	0.0098	330	27	1,370	112	490	39	1,380	112	414	34	67	71

* Analyses made by Hercules Laboratory.
All results based on grab samples collected at 10:00 AM on dates indicated.

Hercules Naval Stores Survey
United States Public Health Service

Special Phenol Study, Summary Sheet

Source	Lab. No.	Date 1951	Average Discharge GPM	Average Discharge MGD	Phenols ppm	Phenols lbs/day
Main plant effluent	115	3/5	3,040	4.37	0.4	14.6
	121	3/6	3,370	4.85	0.4	16.2
	125	3/7	3,320	4.77	0.3	11.9
MEAN			3,240	4.66	0.4	14.2

Above results based on 8-hour composite samples (8 AM - 3 PM)

Source	Lab. No.	Date 1951	Average Discharge GPM	Average Discharge MGD	Phenols ppm	Phenols lbs/day
Synthetics plant	114	3/5	445	0.641	0.3	1.6
	120	3/6	395	0.569	0.2	1.1
	124	3/7	415	0.598	0.1	0.5
MEAN			418	0.600	0.2	1.1

Above results based on 8-hour composite samples (8 AM - 3 PM)

Source	Lab. No.	Date 1951	Time Sample Collected	Average Discharge gal/day	Phenols ppm	Phenols lbs/day
Isomerization wash water, first wash	184	3/21	6 PM	400	3,120	10.4
	209	3/23	8 PM	400	1,730	5.8
	232	3/31	10 PM	400	2,250	7.5
	241	4/2	12 Mid.	400	1,000	3.3
	257	4/5	4 AM	400	1,740	5.8
MEAN				400	1,970	6.6

Above results based on grab samples collected at indicated time.

SAMPLE POINT Main Plant Effluent

Hercules Naval Stores Survey
Results of Special Studies
Summary Sheet
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Lab. No.	Date 1951	Sample Period	Average Discharge			BOD, 5-day, 20°C.			Oxygen Consumed ppm	pH	Acidity or Alkalinity ppm	Ether-Extractable Material		
			GPM	MG Sample Period	Total MGD	ppm	Lbs. Sample Period	Total lbs./ day				ppm	Lbs. Sample Period	Total lbs./ day
97	3/1	8 AM - 3 PM (8-hr.comp.)	3640	1.75		170	2480		372	8.3	79 T	33	480	
100	3/2	4 PM - 7 AM (16-hr.comp.)	3430	3.30	5.05	170	4670	7150	362	6.0	17 T	65	1790	2270
109	3/2	8 AM - 3 PM (8-hr.comp.)	3460	1.67		370	5130		623	7.3	80 T	23	320	
110	3/3	4 PM - 7 AM (16-hr.comp.)	3420	3.32	4.99	120	3310	8440	266	4.5	59 T	22	610	930
116	3/5	8 AM - 3 PM (8-hr.comp.)	3040	1.46		338	4100		688	8.6	74 T	74	900	
118	3/6	4 PM - 7 AM (16-hr.comp.)	3360	3.24	4.70	166	4470	8570	295	3.7	35 T* 11 M	19	510	1410
	MEAN	(8-hour composites)	3380	1.63		293	3900		561	8.1	78 T	43	570	
	MEAN	(16-hour composites)	3400	3.26		152	4150		308	4.7	38 T	35	970	
	MEAN	(24-hour values)	3390		4.90	200		8050	392		51 T	38		1540

For the purpose of this study, the 24-hour period was taken from 8 AM to 8 AM.

* Total and Mineral Acidity; all other values in this column are Total Alkalinity.

(Continued on next page (95-a)

SAMPLE POINT Main Plant Effluent
(Continued from Page 95)

Hercules Naval Stores Survey
Results of Special Studies
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Lab. No.	Total Solids									Suspended Solids								
	Total			Volatile			Fixed			Total			Volatile			Fixed		
	ppm	Lbs. Sample Period	Total lbs/ day	ppm	Lbs. Sample Period	Total lbs/ day	ppm	Lbs. Sample Period	Total lbs/ day	ppm	Lbs. Sample Period	Total lbs/ day	ppm	Lbs. Sample Period	Total lbs/ day	ppm	Lbs. Sample Period	Total lbs/ day
97	364	5300		132	1920		232	3380		132	1920		46	670		86	1250	
100	223	6130	11430	80	2200	4120	143	3930	7310	84	2300	4220	56	1540	2210	28	760	2010
109	326	4530		134	1860		192	2670		104	1440		58	810		46	640	
110	290	8000	12530	86	2380	4240	204	5620	8290	84	2320	3760	48	1320	2130	36	990	1630
116	396	4820		160	1950		236	2870		112	1360		60	730		52	630	
118	221	5960	10780	70	1880	3830	151	4080	6950	41	1100	2460	33	880	1610	8	220	850
	362	4880		142	1910		220	2970		116	1580		55	740		61	840	
	245	6690		79	2150		166	4540		70	1910		46	1250		24	660	
	284		11570	100		4060	184		7510	85		3490	49		1990	36		1500

Hazelton Naval Stores Survey
Results of Special Studies
Summary Sheet
United States Public Health Service

SAMPLE POINT **Main Plant Effluent**

Lab. No.	Date 1951	Time	Average Discharge GPM	MGD	BOD at 20° C. 5 day ppm	BOD at 20° C. 5 day lbs/day *	Oxygen Consumed ppm	pH	Alkalinity P-Total P-Phenolphthalein ppm	Color ppm	Total Solids: Total ppm	Total Solids: Total lbs/day *	Total Solids: Volatile ppm	Total Solids: Volatile lbs/day *	Total Solids: Fixed ppm	Total Solids: Fixed lbs/day *	Suspended Solids: Total ppm	Suspended Solids: Total lbs/day *	Suspended Solids: Volatile ppm	Suspended Solids: Volatile lbs/day *	Suspended Solids: Fixed ppm	Suspended Solids: Fixed lbs/day *
38	2/20	3 PM	3890	4.45	208	10700	711	7.8	114T 0P	900	444	18450	230	8520	214	7930	62	2300	50	1850	12	450
60	2/22	3 PM	3270	4.70	355	13900	681	10.6	352T 44P	- 900	1170	45800	254	9940	916	35860	116	4540	48	1880	68	2660
113	3/4	12 N	3400	4.97	442	18300	759	11.4	404T 21.5P	-	829	34320	335	13900	494	20420	-	-	-	-	-	-
189	3/30	10 AM	3400	4.97	189	7800	-	7.1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
231	4/2	11 AM	3400	4.97	207	8600	-	6.3	-	-	-	-	-	-	-	-	-	-	-	-	-	-
260	4/3	9 AM	3400	4.97	230	9500	-	8.8	-	-	-	-	-	-	-	-	-	-	-	-	-	-
288	4/4	10 AM	3400	4.97	324	13400	-	7.2	-	-	-	-	-	-	-	-	-	-	-	-	-	-
298	4/5	9 AM	3400	4.97	306	12700	-	7.1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
409	4/18	9 AM	3400	4.97	309	12800	-	6.6	54T 0P	-	-	-	-	-	-	-	-	-	-	-	-	-
413	4/19	10 AM	3400	4.97	428	17500	734	7.8	129T 0P	-	-	-	-	-	-	-	-	-	-	-	-	-
417	4/20	9 AM	3400	4.97	1295	53600	1484	9.3	206T 49P	-	-	-	-	-	-	-	-	-	-	-	-	-
MEAN			3360	4.90	397	16200	896		210T 50P	900	814	32190	293	10790	581	21430	89	3423	49	1860	40	1560
MAXIMUM			3400	4.97	1295	53600	1484	11.4	404T 21.5P	900	1170	45800	335	13900	916	35860	116	4540	56	1880	68	2660
MINIMUM			3090	4.45	189	7800	681	6.3	54T 0P	900	444	18450	230	8520	214	7930	62	2300	48	1850	12	450

* lbs/day values were computed on the results of grab samples and are not absolute 24 hour values.

Main plant effluent diverted through lagoon after 2/28/51.